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FINAL PROGRESS REPORT
RADIATION-RESISTANT ELECTRONICS

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1 January 1963 through 30 September 1963

HARRY DIAMOND LABORATORIES
ARMY MATERIEL COMMAND
WASHINGTON 25, D. C.

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UNITED STATES ARMY MATERIEL COMMAND
HARRY DIAMOND LABORATORIES
WASHINGTON 25, D.C.

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FOR THE COMMANDER:
Approved by

P. E. Landis
Chief, Laboratory 900

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ABSTRACT

This is the fourth and final progress report on a program to develop radiation-resistant electronics. The program was managed by the Harry Diamond Laboratories with the support of the National Aeronautics and Space Administration. In addition to in-house work at HDL, contracts were let with the Philco Corporation to develop thin-film active devices and with the General Electric Company for work on tunneling cathodes. The contractors submitted individual quarterly progress reports and final reports are in preparation.

During the period covered by this report the theoretical description of the field emission process developed earlier has been applied to ten sets of experiment data from several sources. Although the theory could be made to fit the data, question still exists as to the validity of the values that must be assumed for some of the adjustable constants, such as the generally unmeasured film thicknesses. A special report on this theoretical work has been issued separately.

Although further experiments were performed with aluminum-aluminum oxide-aluminum sandwiches, the emphasis of the experiments was changed to gold-silicon monoxide-gold structures since these are amenable to interferometric measurement of the insulator layer. Early experiments with the latter system produced many short circuits. Underlaying the gold electrode with oxidized bismuth, producing a feather edge on this electrode, and annealing the composite structure prior to a carefully controlled silicon monoxide deposition dramatically reduced the incidence of short$_0$ circuits. It is now possible to produce these sandwiches with only 75 Å of silicon monoxide between the gold layers without short circuits. A large number of thin film diodes were made and their electrical behavior examined. Unfortunately for the practical objectives of this research, the electrical behavior of these thin film structures was complicated by a variety of effects not directly related to the quantum mechanical tunneling hoped for. In fact in these experiments tunneling was seldom encountered.

Work was continued on the preparation of thin-film, aluminum-cadmium sulfide-gold diodes for possible collector structures. When these were subjected to an appropriate heat treatment they developed pronounced rectifying characteristics thereby reinforcing the idea that a successful thin-film collector could be made in this manner.

Since this is the final report of the program on Radiation-Resistant Electronics, managed by HDL in behalf of NASA, a review of the results of the two industrial research contracts, which ran concurrently with the in-house effort, is also included as well as a historical survey of published research in this field appearing during the period covered by this report. It is concluded that while a hot-electron active device based upon quantum mechanical tunneling is by no means out of the question, significant problems in the fabrication of such a device still remain to be overcome.

1. INTRODUCTION

This is the fourth and final progress report describing work on radiation-resistant electronics, which was supported by the National Aeronautics and Space Administration. The program consisted of an in-house effort at the Harry Diamond Laboratories and two contracted programs, one at the Philco Corporation and another at the General Electric Company. The program had as its goal, the development of radiation-resistant electronic devices utilizing tunneling or other conduction phenomena in thin-film structures. This report gives an account of the research at HDL; the contractors' researches are reported in their own reports.

2. PROGRESS - THEORETICAL

2.1 Analysis of Field Emission according to the Theory of Murphy and Good

During the previous report period, a theoretical description of the field emission process that occurs in thin-film, metal-insulator-metal sandwiches had been developed. The analysis included the effects of image forces in a consideration of combined thermonic emission and quantum mechanical tunneling. During the period reported here, data on tunneling currents were collected from ten sources, viz: one set of data on aluminum-aluminum oxide-aluminum and one set of data on gold-silicon monoxide-gold were from HDL experiments and the reminder were from the literature. Of the latter, one set of data was on beryllium oxide, the rest were on aluminum oxide. The theoretical curves could be made to fit the data in most cases, but the physical significance of some of the values attached to the adjustable theoretical parameters is questionable. For example, it is frequently necessary to assume film thicknesses greater than that estimated by the experimenter. A detailed report of this work has been issued separately rather than within this series of progress reports (ref 1), and a slightly condensed version has been accepted for publication in the Transactions of the I.E.E.E. for Component Parts.

3. PROGRESS - EXPERIMENTAL

3.1 Vacuum Technology

All the experiments performed to date, including those reported in this report, below, have been performed in conventional high-vacuum systems, which can provide an ultimate vacuum no better than 10^{-7} torr. In discussions with experts in the field of thin-film research, it has been made manifest that superior results can be obtained by going to ultrahigh vacuum, i. e., to at least 10^{-9} torr. Accordingly we have concluded that future experimentation in this connection will require the use of ultrahigh-vacuum equipment if there is to be a reasonable chance for success.

3.2 Diode Structures

Experiments were continued to obtain data on tunneling currents in thin-film structures consisting of two metal films separated by a thin insulating film. In a continuation of earlier work, further sandwiches of aluminum-aluminum oxide-aluminum were prepared. Although this system is theoretically a promising one, it has two practical drawbacks as far as the present work is concerned. In the first place, it is exceedingly difficult to measure the oxide thickness accurately. In the second place, unless the first metallic aluminum electrode is vacuum deposited in ultrahigh vacuum, the oxide layer which is grown thermally will not have good insulating properties. In the work reported here, no ultrahigh-vacuum facilities were available so that it was not surprising that the greater portion of our thin-film structures exhibited short circuits. Nevertheless it was possible to observe field emission in a limited number of cases and an example of the results obtained is given in figure 1. These d-c data have been examined theoretically, and an internal work function, ϕ, of 0.6 eV has been obtained for the aluminum-aluminum oxide interface, as well as a dielectric constant, K, of about 3 for the oxide. This analysis has been given in detail elsewhere (ref 1).

Because of the practical limitations enumerated above, it was decided to conduct subsequent experiments with gold-silicon monoxide-gold structures. Here the thickness of the insulating film could be measured accurately by the multiple beam interferometer and there was no apparent requirement for ultrahigh vacuum. At the outset the frequent occurrence of short circuits in such sandwiches was a considerable impediment to progress. They were frequent even with silicon monoxide layers [redacted] as thick as 800 Å. These short circuits were thought to have their origin in the characteristically granular nature of vacuum-deposited gold films, which present protrusions capable of penetrating the thin silicon monoxide layers. Since it had been recorded in the literature (ref 2, 3) that films of gold sputtered onto bismuth oxide are strikingly smooth, it seemed logical to apply a related procedure to the vacuum-deposited gold. Accordingly, sandwiches were made by first vacuum depositing a film of bismuth, oxidizing the exposed surface thereof, and then vacuum depositing the gold onto the bismuth oxide. The composite gold electrode was then annealed at 100°C for one hour.

A second change of procedure was intended to remove the sharp edges from the vacuum-deposited electrode described above. Thus the mask used in these depositions was located 1/8 in. in front of the substrate rather than in direct contact with it so that the shadow cast by the mask was diffused at the edges. The electrodes prepared in this way have been shown to have gently sloping edges, which minimize any mechanical stresses in their vicinity within the insulator. Also the silicon monoxide source temperatures were carefully controlled so that the internal stresses of thermal origin were minimized (ref 4). These changes evidently had a benefical effect and many small area devices,

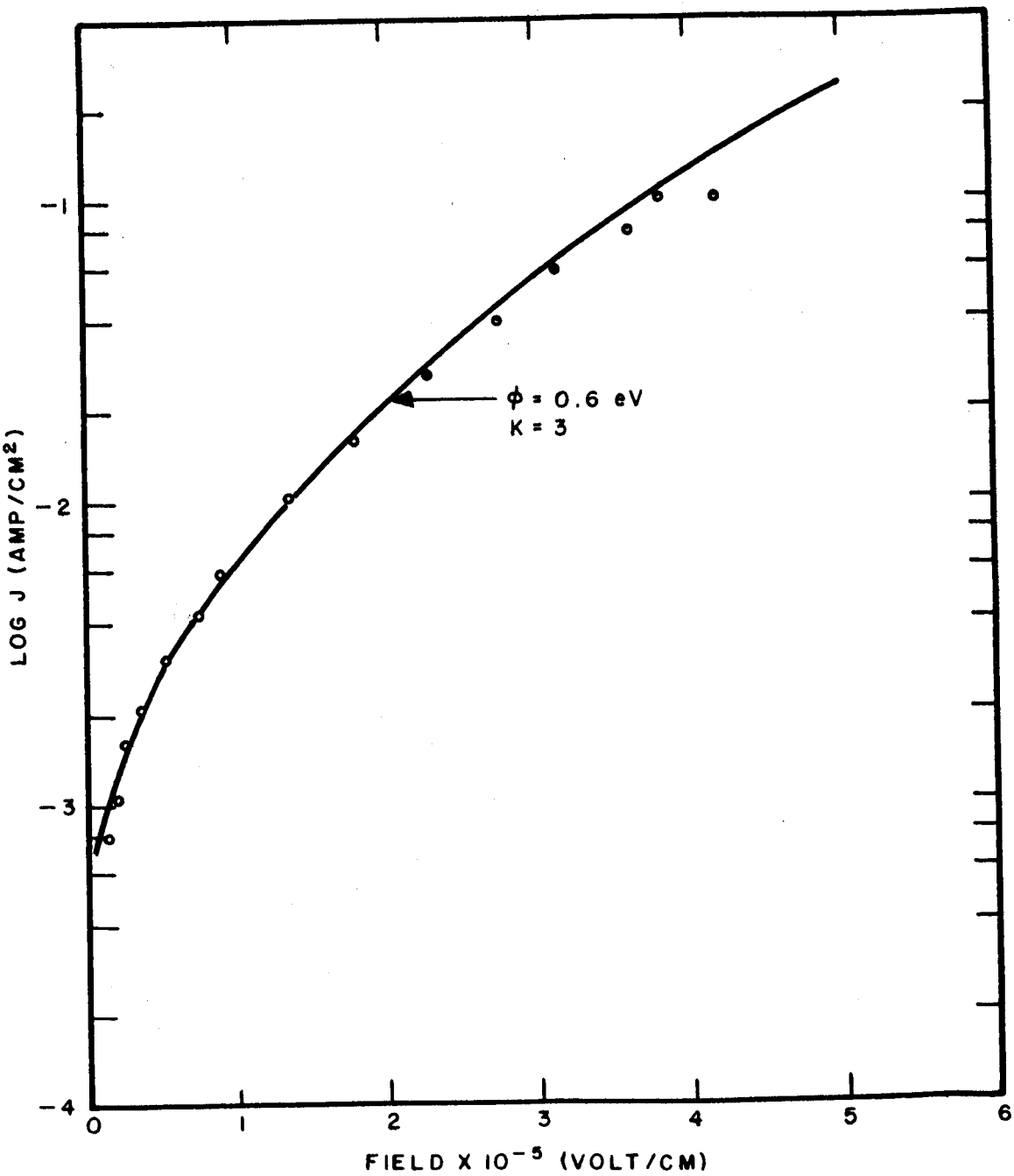

Figure 1. Field emission characteristics of aluminum-aluminum oxide-aluminum thin-film diode. Points are experimental, curve is theoretical.

i.e., active area 0.25 mm^2, were then made free of short circuits, although on larger area devices, i.e., active area 4mm^2, short circuits still occurred. This improvement permitted a reduction of the insulator thickness to as little as 75 Å without a serious incidence of short circuits on the small devices. When the insulator thickness was further reduced to 50 Å, all units were again short circuited. The possibility exists that comparable gold electrodes made by a sputtering process would be even smoother and might permit insulator thicknesses below 75 Å to be useful. From a theoretical point of view such films would be attactive.

During the course of this work, 240 individual diodes were fabricated in arrays of 12 to a slide, each array presenting three different areas. The overwhelming majority of the diodes that were not short circuited showed complicating effects that were not directly connected with the field emission process. These fell roughly into two classes. The first comprised diodes that showed a low initial resistance, usually a few ohms; the second comprised diodes that showed a high initial resistance, a megohm or higher.

Diodes of the first class were characterized by an unstable current-voltage curve, which underwent a permanent change when a voltage in excess of a certain threshold value, peculiar to the specimen under test, was exceeded. After the application of voltages larger than the threshold, the voltage-current characteristic became more stable and corresponded to an even lower resistance than before. Apparently these diodes were undergoing an electrical forming process. After being formed, some of these showed nonlinear current-voltage curves typical of a field emission process. An example is shown in figure 2 in which the experimental d-c points are fitted to the theoretical curve for combined thermionic and tunnel emission. This correlation is explained in the previous reference (ref 1), and through it a value of 0.5 eV was obtained for the internal work function, ϕ, and a value of 4 for the relative dielectric constant, K, of the silicon monoxide. Some of the diodes, when formed, exhibited a linear voltage-current curve and these remain an enigma.

Those diodes that initially presented a high resistance, exhibited a different behavior, which may be summarized as follows: the diode as usually fabricated presented an extremely high resistance as the applied voltage pulse of the curve tracer (i.e., rectified 60-cps ac) was increased from zero to a certain threshold value. This would be a few volts, the exact value depending upon the diode in question. Upon exceeding the threshold, the diode switched to a low resistance condition and the current increased, while the voltage dropped due to the action of a series, current-limiting resistance. Now the current-voltage curve was linear. Upon increasing the current further, a current was finally reached at which the diode switched back to its high resistance state or to an intermediate state also of high resistance. This cycle of switching could be repeated over and over again (but not

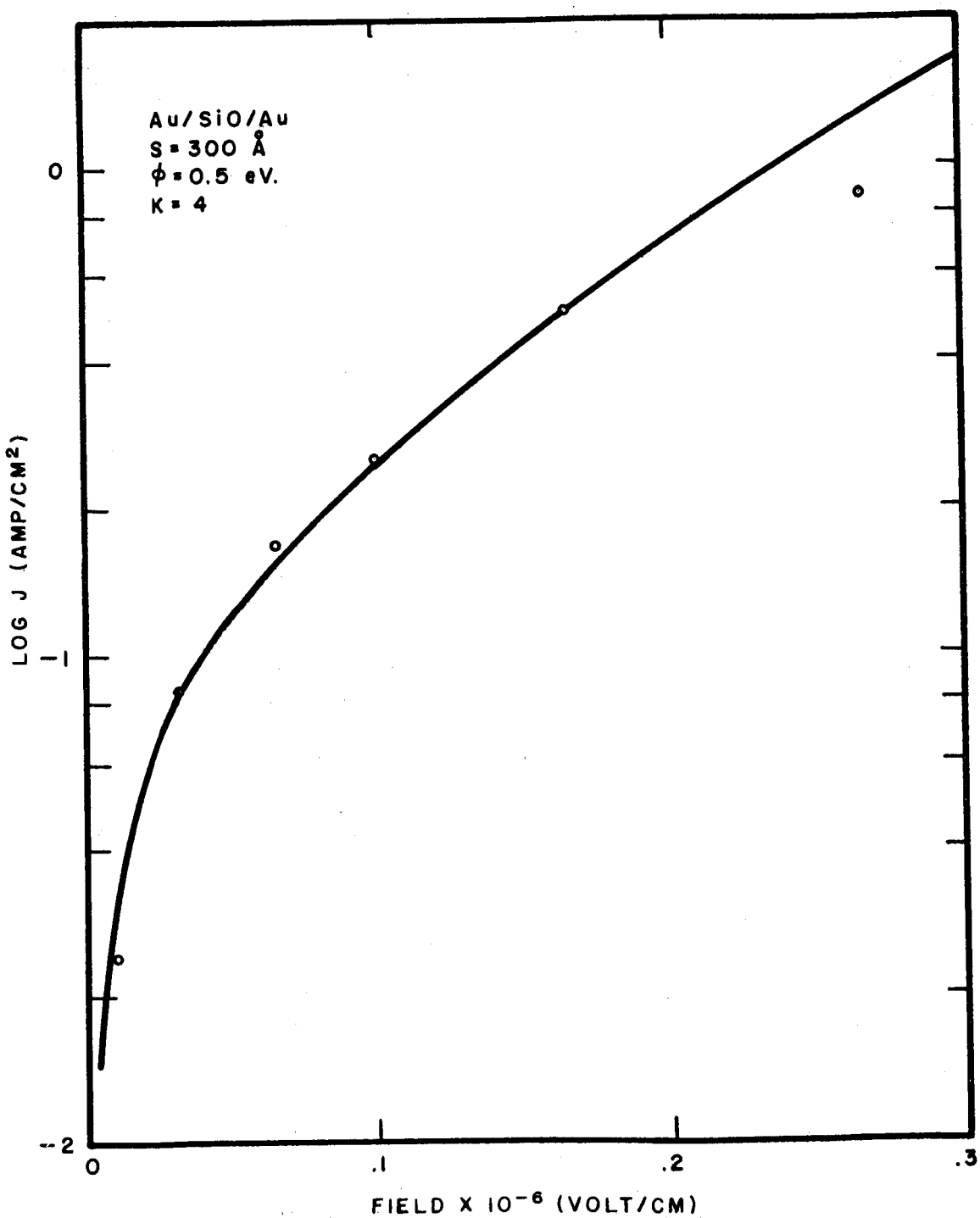

Figure 2. Field emission characteristics of gold-silicon monoxide-gold thin-film diode. Points are experimental, curve is theoretical.

indefinitely); it evidently did not matter whether the counter electrode was positive or negative with respect to the first deposited electrode. The same behavior occurred when the polarity of the applied voltage pulse was reversed. For example, let the diode be switched on by a voltage pulse in a particular direction. If the current was then reversed, the diode would switch to the high resistance state when a current threshold in the reverse direction had been exceeded. The switching from high resistance to low appeared to be voltage controlled and that from low resistance to high appeared to be current controlled, as will become more evident farther on.

Certain other aspects of the behavior outlined above appeared when the switching phenomena was examined in detail. For example, with most diodes in room ambient a spontaneous switching often occurred in which the diode would switch back and forth between the high and low impedance conditions sporadically. This behavior was voltage dependent. Desiccating such diodes in a stream of dry nitrogen eliminated this effect, so doubtless it had its origin in electrolytic processes within the insulator. The normal (i.e., not spontaneous) switching cycle from high to low impedance could be triggered by light from a fluorescent lamp, although voltage transients from the lamp could possibly be a factor here as well. The spontaneous switching was similarly effected, and in fact could be accomplished with lower applied voltages than in the normal case.

The forming and switching effects uncovered in these experiments appear to be generally observed in the gold-silicon monoxide-gold diodes made in our laboratories. To our knowledge they have not been observed with these materials elsewhere and others have made similar diodes but with generally thicker films without observing these effects (ref 5). Accordingly it appeared worthwhile to examine these effects further and to try to understand their origins with a view toward eliminating them.

Returning to the low resistance diodes, an examination of the voltages required for the forming process as a function of the thickness of the insulating layer, measured interferometrically on a "control" microscopic slide, led immediately to the conclusion that the voltages and thicknesses were basically linearly related (fig. 3). The forming took place in an electric field of about 2.5×10^5 V/cm as calculated from the slope of the best straight line. This value of field strength is larger than that associated with the drift of relatively mobile ions in solids, for example 6×10^4 V/cm in the case of lithium drift in silicon (ref 6). This suggests that a related cause might be operating here. The actual values of the formed and unformed resistances varied considerably from diode to diode, but typical performance would be demonstrated by a diode with an initial resistance of 20 ohms and a formed resistance of 10 ohms. The forming process represents a permanent change in the diode, and once a diode is formed, its

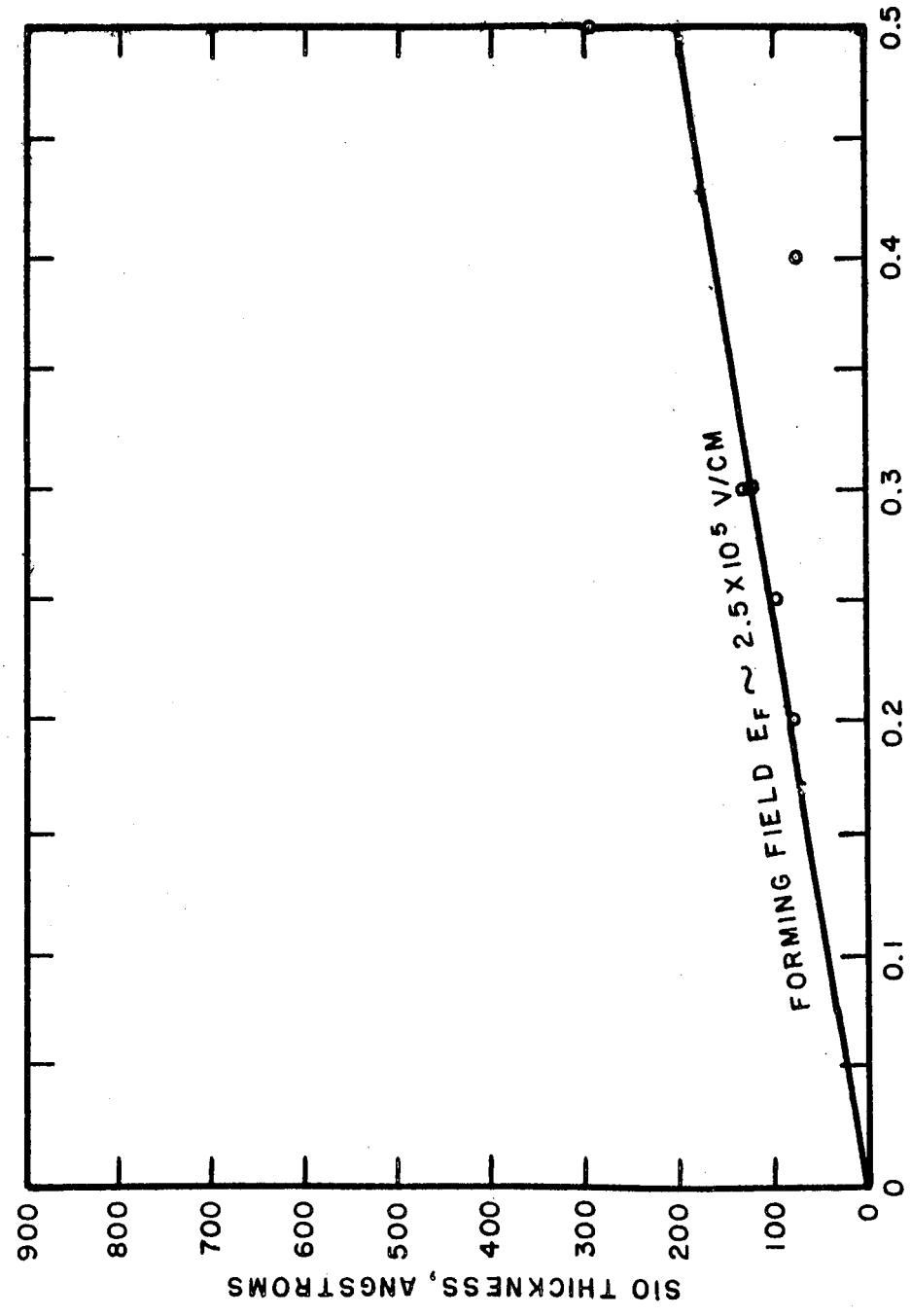

Figure 3. Forming voltage versus thickness for low resistance diodes.

low resistance persists irrespective of the direction of the applied voltage. Of the diodes formed, many gave ohmic behavior after forming and were obviously not tunnel emitters. The remainder were nonlinear but generally not sufficiently stable electrically over several decades of current to be useful. Only a very few appeared to be field emitting.

Examination of the data obtained from the high resistance diodes showed no obvious correlation between insulating layer thickness, threshold voltage for switching, and active area of the diode delineated by the overlap of the electrodes. In these experiments the areas were 4 mm^2, 1 mm^2, and 0.25 mm^2. Although the forming phenomenon discussed above was never observed on diodes of the smallest area, the switching phenomenon was observed to occur on diodes of all areas. Also, though the high resistance diodes could apparently be switched on and off repeatedly, it was soon noticed that diodes that had been switched many times changed color over the active area, from gold to pink. Microscopic examination during switching showed the progressive formation of granules from what had been a smooth gold film. Eventually the granulation proceeded so far that electrical conductivity along the gold film was destroyed. Random scintillations over the active area were also observed in the dark, which seemed to correspond to the spontaneous switching seen in the curve tracer. In a typical example, scintillations of approximately white light appeared at a threshold of 19 V for a 750 Å silicon monoxide insulating layer. As the voltage across the film was increased to 25 V, the light changed to blue. Here the luminescence gradually moved to the edges of the diode active area and ultimately resulted in the extensive vaporization of the metal films until they were discontinuous in this region. When the microscope slide, which served as the substrate for the diodes was turned over, the same effects were observed at the glass-metal interface.

A diode that had not yet been switched was examined at 750X for defects resulting from fabrication or aging that might start the switching. Only a few random dark spots about 1 μ in diameter were visible within the active area, but dark whiskers about 10 μ in length were seen at the edges. These observations are significant for interpreting the series of photomicrographs of the medium area diode (1 mm^2 active area) after several stages of switching shown in figures 4 to 7. Figure 4 shows a diode active area before switching with relatively smooth films apparent. The edges are diffuse, because of the taper produced in the cross section by the masking procedure described earlier, and present a rather spongy appearance, becoming transparent as one moves outward from the center. Figure 5 shows the results of preliminary switching at low voltages (ca. 1 volt). Slight granulation appears in the center of the active area, but the main attack on the electrode is at the edge. Figure 6 shows a diode after extensive switching, with scintillations observed at voltages up to 25 V, at which point one lead to one of the metallic film electrodes burned out. Granular structure now covers the entire active area, and although not clearly shown in the photomicrograph, whiskers populate the edges. Figure 7 shows the diode after even more switching and here the effects are even more extreme featuring a finer, denser granulation.

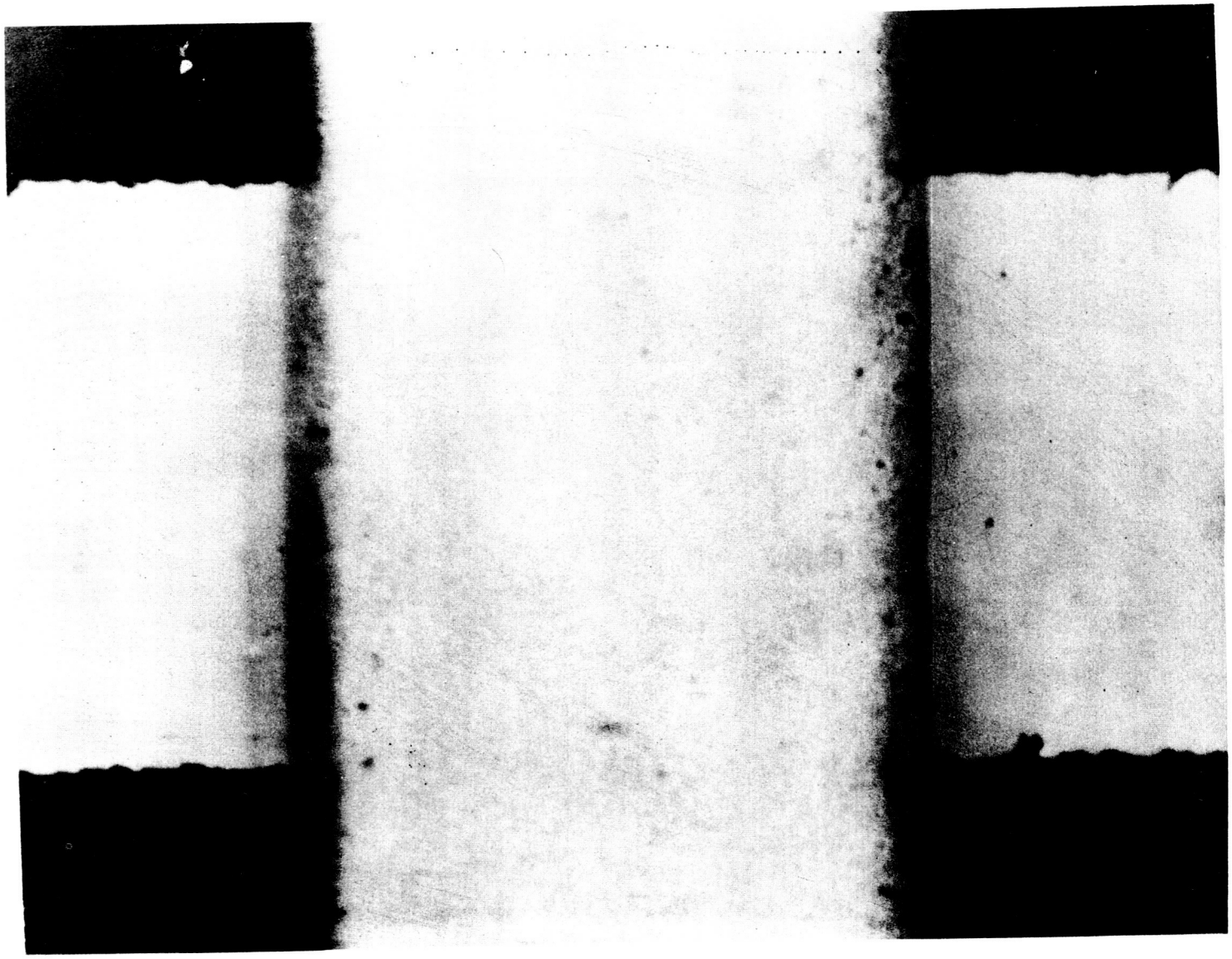

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Figure 4. Photomicrograph of active area of thin-film, gold - silicon monoxide - gold diode before switching.

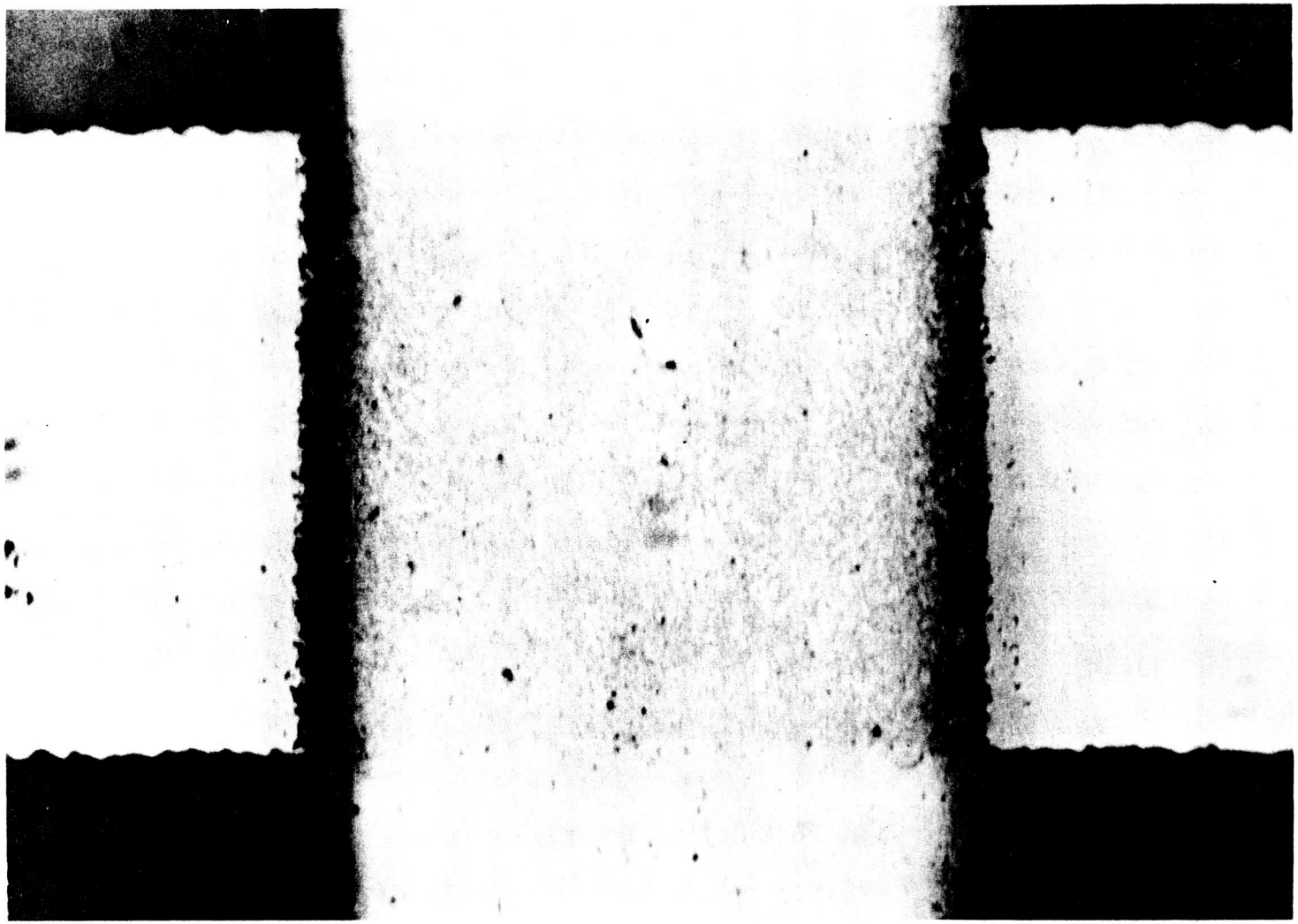

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Figure 5. Photomicrograph of active area of thin-film, gold - silicon monoxide - gold diode after an interval of repeated spontaneous switching at low voltage (approx. 1 volt).

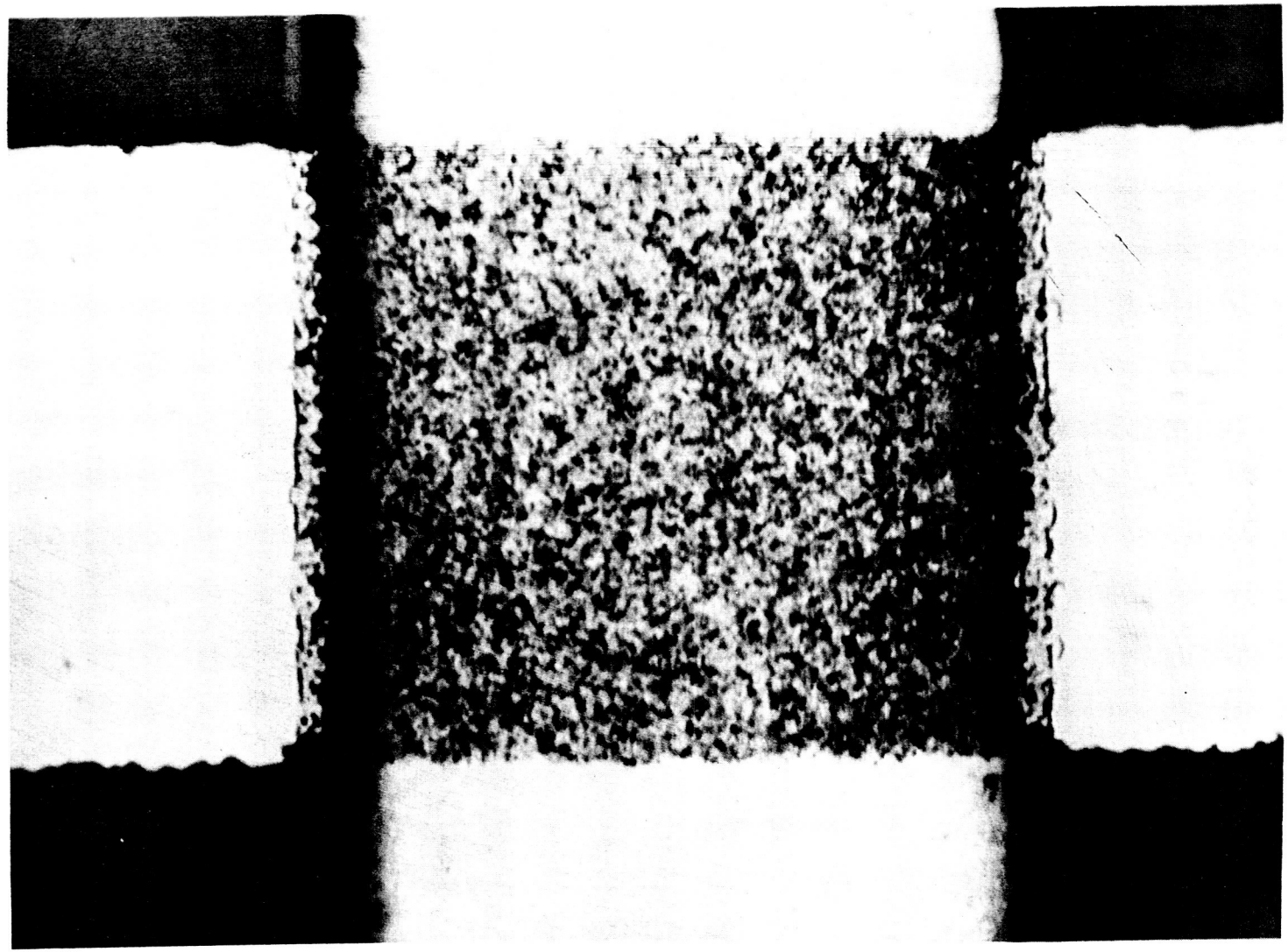

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Figure 6. Photomicrograph of diode after a second interval of repeated, spontaneous switching at higher voltages (up to 25v).

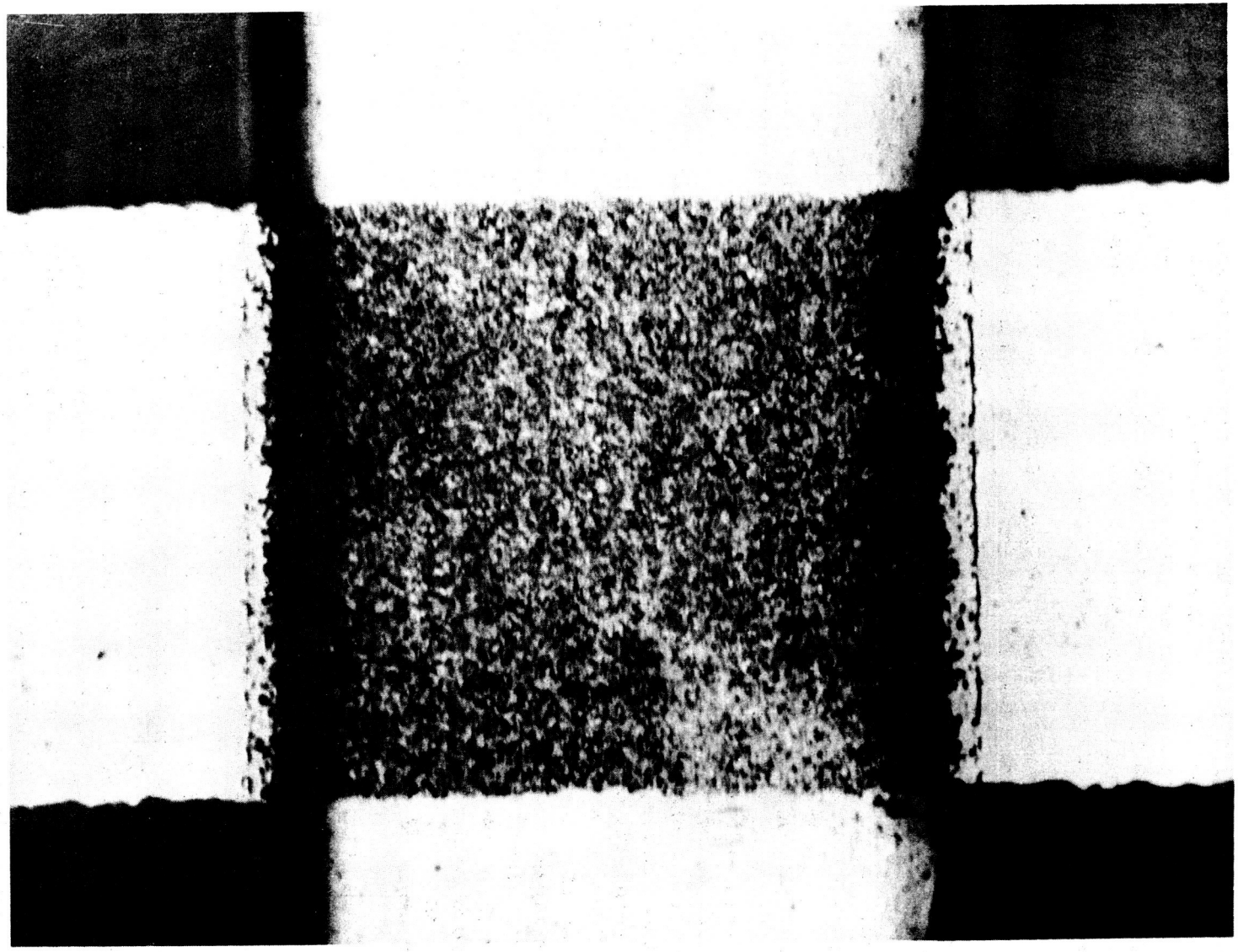

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Figure 7. Photomicrograph of diode switched almost to destruction.

This discussion is reminiscent of work done at Stanford University in connection with the system nickel-nickel oxide-nickel. A similar switching phenomenon appears there and it was attributed to the formation, under the influence of the applied electric field, of filamentary metal bridges between the electrodes (ref 7). This would switch the diode on, and high current passed at a later time would thermally destroy the filament to switch the diode off. Such a mechanism applied to the gold-silicon monoxide-gold diodes discussed here would explain many of the effects observed.

In a recent paper, Pollack, Freitag, and Morris (ref 8) have observed somewhat different effects in thin film diodes that may be related to the phenomena observed here. In their work, lead-aluminum oxide-lead diodes were changed from very high to low resistances by virtue of a positive ionic space charge, which drifted toward the cathode in an applied field of 3×10^6 V/cm. The effect was unidirectional, i.e., the diode was polarized to conduct in one direction only--a polarizing voltage in the opposite direction was required to insure high reverse conductivity. Because of the very large resistance changes observed by these authors, their observations are most easily compared with the behavior of our high resistance diodes. The latter differed significantly in that they formed bidirectionally, i.e., conduction in both directions was enhanced by a single poling in either direction. Whereas the diodes of Pollack et al had nonlinear current-voltage curves, our diodes had linear current-voltage curves, symmetrical about the origin at all times. This symmetry is shown in the curves of figure 8, which have been made to go through the origin by the application of a small d-c bias to the diode under test in the curve tracer. This figure shows several states of intermediate resistance for a single diode, which were induced by the switching of a fluorescent lamp near the test specimen. The diodes of Pollack et al gave rise to measurable after-currents upon the spontaneous relaxation of polarization when the voltage was removed. Our diodes produced no such after-currents.

3.3 Organic Thin Films

Following a report in the literature (ref 9) to the effect that tunnel-emitting sandwiches could be made with polymeric insulating layers, a small effort was devoted to the formation of thin films of polyvinyl formal. Organic thin films may be deposited onto a substrate in a variety of ways, but the simplest method, and that chosen for these preliminary experiments, consisted in preparing a dilute solution of a high molecular-weight grade of polyvinyl formal in chloroform, dipping a suitably cleaned microscope slide into this solution, and then gradually withdrawing the slide from the solution, leaving its surface coated with a liquid film of the polymer solution. Upon evaporation of the solvent, a thin solid polymer film remained. Reasonable precautions were taken in performing these experiments. The slides were withdrawn from the solution by a motor-driven device, which

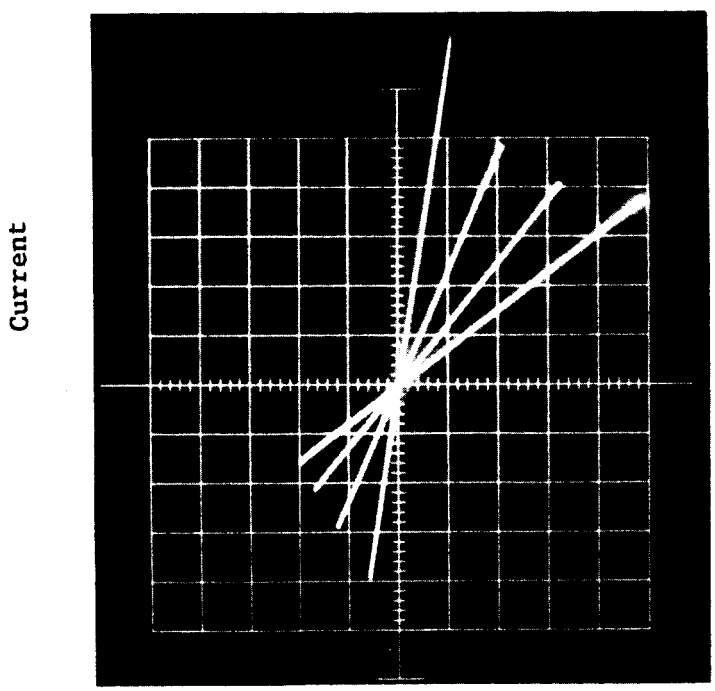

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Figure 8. Current-voltage characteristic of a switching diode showing several bidirectional resistance states. Vertical scale = 20μ A/div. Horizontal scale = 0.2 V/div.

provided uniform withdrawal rates of from 25 to 375 mm per minute. The solution was placed in a glass cylinder 200 mm tall and 63 mm in diameter filled to only about 75 mm from the bottom so that the liquid surface was somewhat protected from air turbulence. The solutions were pressure filtered into the cylinder through glass frit filters immediately before use, to remove dust. Within the range of withdrawal rates quoted, the film thickness was at best a slowly varying function of rate. In most of our experiments, a rate of 75 mm per minute was used. The films were allowed to dry for several hours in a clean air hood and were then baked overnight between 40° and 50°C to drive off the bulk of the retained solvent.

The films made in the manner described presented a uniformly good appearance although, despite the precautions taken, dust inclusion was still a problem. The film thickness was almost entirely determined by the concentration of the polymer in the solution; films approximately 100 Å thick were deposited from solutions containing 1/2 gram of polyvinyl formal in 100 ml of chloroform solution. However at any one concentration, the spread of thicknesses was considerable and could range from 50 to 100 Å at the concentration quoted above. The cause of this variability has not been investigated. Attempts to measure the thickness of these films interferometrically revealed a not entirely unexpected difficulty. The leading edge of the films showed the formation of a lip, which protruded well above the top of the film that followed it in formation. A typical example is shown in the upper interferogram of figure 9. Here, the lip rises 671 Å above the substrate, whereas immediately after the lip is formed, the film commences with a thickness of 255 Å. The lip has its origin in a slight enrichment of the solution caused by solvent evaporation from the surface prior to the withdrawal of the microscope slide-substrate. In the first experiments, the film thickness was measured between the substrate and the opposite side of the lip, but close examination of many interferograms indicated that the film thickness, after passing through a minimum after the lip, seemed to increase gradually to a somewhat larger value further on. To examine this possibility, a mechanical device was built that would cut a notch sharply through the film but not through the substrate. The device, shown in figure 10, employed an aluminum blade to make the cut. The blade was softer than the glass, but harder than the film. The results of such a cut through the film discussed above are shown in the lower interferogram of figure 9. Here it is apparent that a well defined notch was indeed obtained, but that the film thickness now had increased to 557 Å. Needless to say, a variety of experiments was performed to make certain that this cut did not extend into the substrate. There is now no reason to believe that the notching method does not yield a reliable film thickness or that the film is not thicker at its center than at its leading edge.

Several groups of diodes were made with vacuum-deposited gold electrodes and polyvinyl formal films roughly 100 Å thick. In making these diodes the same procedures were used to insure a smooth lower

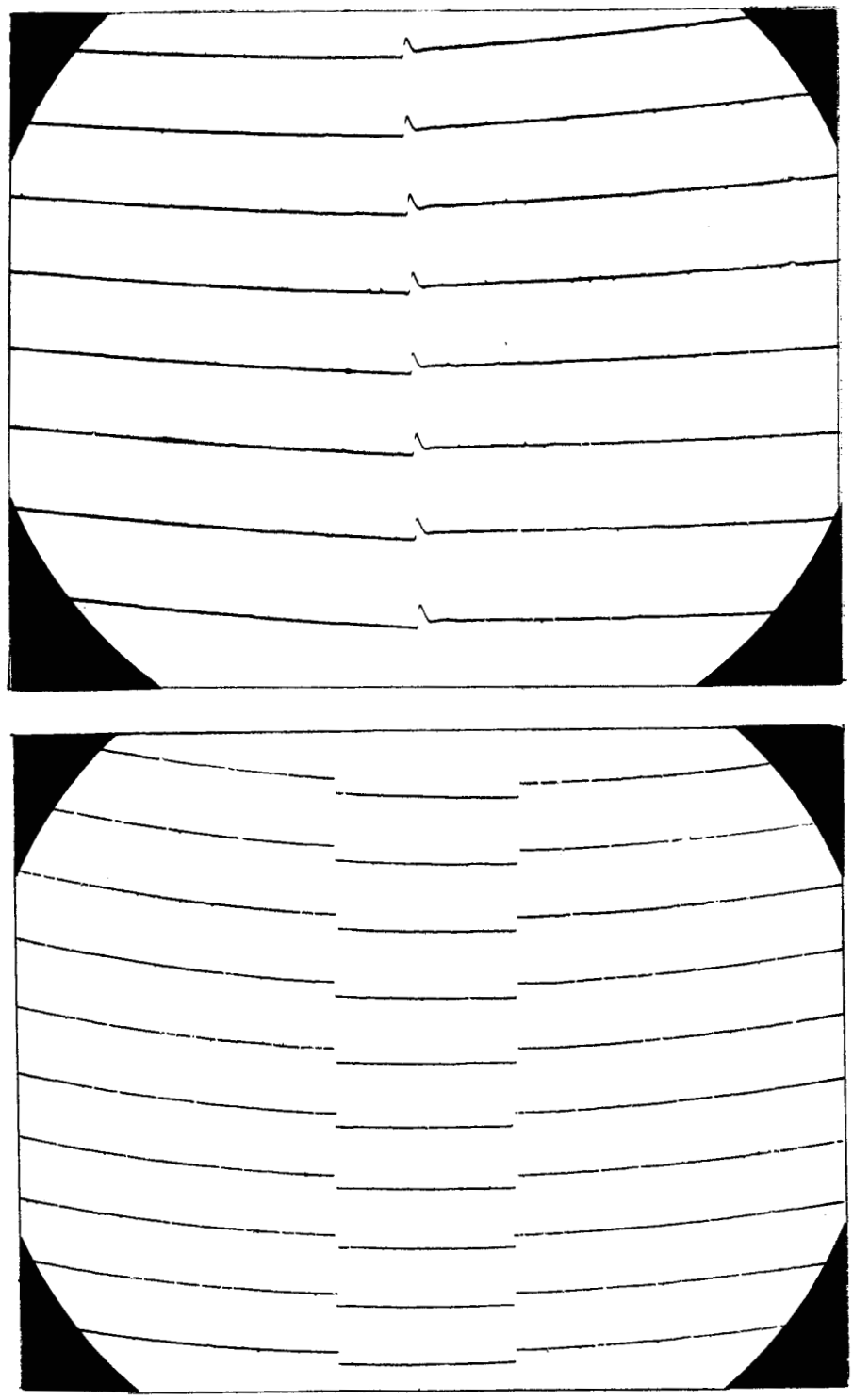

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Figure 9. Multiple beam interferograms of organic films deposited from solution. Upper: leading edge of film. Lower: notch cut through main part of film.

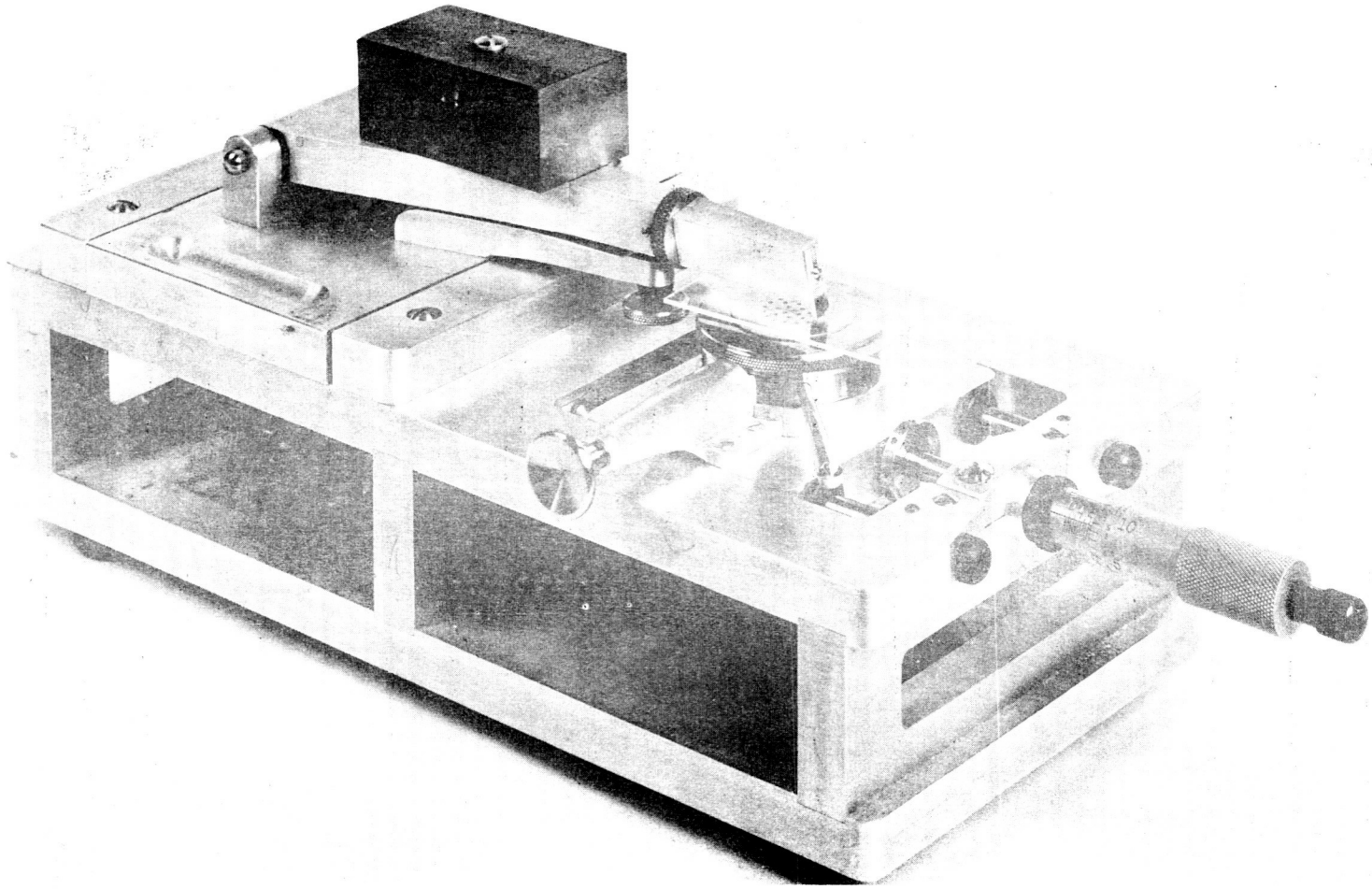

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Figure 10. Notching machine for thin organic films.

electrode as were used in making the gold-silicon monoxide-gold diodes described earlier (sect 3.2). Nevertheless these diodes all presented short circuits. Accordingly the electrical problem was examined in greater detail. Starting with a fresh sample of polyvinyl formal (i.e., Formvar 15/95S), four groups of eight diodes each were prepared, with thicknesses of polymer ranging from about 50 Å to 550 Å from group to group. All of the diodes made in these experiments exhibited short circuits. It was then thought that a likely source of the trouble resided in the choice of chloroform as a solvent, inasmuch as the more successful diodes reported in the literature had been prepared from dioxane solutions. The reasoning behind this was elementary: chloroform being a chlorinated solvent and structurally dissimilar to the oxygenated polymer could well be expected to form a solution in which the polymer chains were tightly coiled (i.e., low polymer-solvent interaction) and likely to form a discontinuous film at these thicknesses. Dioxane, being an oxygenated solvent would be more likely to form a solution with more extended chains. Such solvent effects are well known in polymer chemistry (ref 10). Accordingly subsequent experiments were performed with dioxane solutions. A solution 1.6 gm of polyvinyl formal in 100 ml dioxane gave a 1000 Å insulating film at a withdrawal rate of 100 mm per minute. Four groups of eight diodes each were prepared with thicknesses of polymer ranging from about 125 Å to 1000 Å from group to group. Despite the change in solvent, all of these diodes exhibited short circuits. Accordingly a final experiment was performed in which the rate of withdrawal of the substrate from the dioxane solution was drastically reduced, to 17 mm per minute, other conditions remaining unchanged. The 32 diodes made in this manner also exhibited short circuits. One is reluctantly forced to the conclusion that short circuits are characteristic of gold-polyvinyl formal-gold diodes prepared under the conditions of these experiments.

The diodes reported in the literature had, in fact, been prepared with a chromium lower electrode, a polyvinyl formal insulating layer, and a gold counter electrode. We have avoided the use of the chromium because chromium is known to grow an oxide layer, and this may make an important and unknown contribution to the insulating layer. However, in view of our inability to develop satisfactory insulating films in the experiments above, it was desirable to examine the chromium-polyvinyl formal-gold system. Accordingly one group of eight diodes of the latter composition were prepared with 125 Å polyvinyl formal layers. All of these diodes exhibited measurable resistances and, in fact, some of them exhibited rather high resistances. Four of the eight diodes were measured and the following resistances obtained: 2,400 ohms, 3,600 ohms, 4,800 ohms, and 10,000 ohms. The voltage-current curves for these diodes were linear (in contrast to those reported in ref 9) so that tunneling and/or thermionic emission was not the conduction mechanism involved here. Strangely enough the diodes of smallest area exhibited the lowest resistances, and those of largest area exhibited the largest resistances. This is contrary to what one would expect. If one corrects the resistances given above to the resistances that would

be exhibited by identical diodes, one square centimeter in area (i.e., to a "specific diode resistance"), one obtains the respective values 20.6 ohms, 75.6 ohms, 146 ohms, and 382 ohms.

Clearly diodes made with chromium lower electrodes are different from those made with gold lower electrodes. The short circuits obtained in the latter, and not in the former, may be explained on the basis of gold from the counter electrode penetrating the polymer film in both cases and the oxide layer on the chromium preventing short circuits in the former case. This does not explain why our current-voltage characteristics are linear whereas those of the literature are nonlinear. Neither does it explain the wide variation of specific diode resistances obtained with the chromium-polymer-gold diodes.

In view of the difficulties experienced with polymer films deposited from solution, a parallel approach to this problem was initiated whereby polymer films could be deposited photochemically from a vapor phase consisting predominantly of monomer (ref 11). Such an approach is attractive because it is recognized that when polymer films are formed from solution, they experience a large decrease in volume during the evaporation of the solvent. If the polymer network cannot completely accommodate all of this shrinkage, a somewhat porous structure will almost certainly result. A polymerization from the vapor phase would obviate this difficulty because the polymer film could, by suitable choice of monomer, be made to form in situ in the absence of a solvent phase. Accordingly, a gas-handling system was constructed in which experiments on the photochemical deposition of polymer films could be performed during the period reported here. An ASA code H4AB mercury vapor lamp was used as a source of radiant energy. This is a 100-watt lamp, not of the high pressure variety, with an inner quartz jacket and an outer glass jacket. The latter was removed so that the spectral lines in the ultraviolet were available. The lamp was located at one focus of a cylindrical aluminum mirror of elliptical cross section. A quartz reaction tube was placed at the other focus. The major axis of the ellipse was 300 mm long, its minor axis 200 mm long, and the height of the cylinder was 200 mm. The reaction tube was 38 mm in diameter. This part of the system was inclosed in a light-tight box. Methyl methacrylate was chosen as a suitable monomer for the first experiments and the system was put into operation just before the close of the period reported here. The very few runs that have been made do indeed show that polymer films, approximately 100 Å thick can be formed on glass substrates. However we have not had time enough to study the process in detail and the films made so far have not been of the best quality. In particular, no attempt has yet been made to separate the near ultraviolet spectral line at 2537 Å, useful in polymerization, from the line at 1849 Å, which is known to degrade polymers. Consequently no meaningful electrical properties have yet been measured on such films in our laboratories. Clearly there are many further experiments that could usefully be done in connection with polymer films.

3.4 Film-Thickness and Electrical Measurements

The multiple beam interferometer for measuring the thickness of thin solid films was largely completed during the report period preceding the present one and has been in continual use since then. Some refinements have been made in this instrument, which have improved its performance. The stage interferometer has been completely rebuilt along the lines suggested in the previous report and in its present form is shown in figure 11. The optical flat is held in a rigid yoke. It is guided by the smooth bosses of the three differential screws, which pass through matching holes in the yokes as the flat is brought in contact with the specimen. When contact is made, the yoke is secured to the differential adjusting screws by nylon-tipped set screws. The final adjustment is made by the differential screws, shown in cross section in figure 12. In this design the motion of the boss relative to the base is controlled by the common rotation of a 4 - 48 thread through the former and a 2 - 56 thread into the latter. The differential motion so produced corresponds to almost exactly 0.003 in. per turn. A pin in the assembly prevents the boss from rotating when a yoke is not attached. The knurled heads or caps are keyed to the double-threaded shaft and rise and fall with the boss rather than the shaft.

As is shown in figure 11, the stage interferometer is presently equipped with a conventional 2-in. diameter optical flat, which has been given a highly reflective dielectric coating. An attachment is also available whereby this large flat can be replaced by a small optical flat, similarly coated, and cut from a flat area of a microscope slide. This flat is only 9/16 in. in diameter and assures a close approach to the specimen under examination, even if the latter is somewhat warped, as sometimes happens. In this event the large flat often cannot be close enough to the specimen for formation of the sharpest interference fringes. Because of the somewhat smoother surface of the fire polished flat compared with that of the large mechanically polished flat, somewhat smoother interference fringes are obtained with it than with the larger flat.

The small flats are cut from microscope slides, precoated to our specifications by an outside concern. This arrangement avoids having the contractor handle very small pieces of glass in his coating operation, but it also requires delicate handling of the coated slides during the cutting of the flat portions. Equipment has been constructed for cutting the small optical flats with minimum damage to the dielectric coating. In this equipment, shown in figure 13, the microscope slide is held between two opposed, rubber-faced platens and rotated slowly beneath the small nozzle of a grit blasting machine. After the circular section is cut, the platens are carefully separated and the flat is immersed in a beaker of methylene chloride. Some of the grit from the cutting operation is unavoidably attracted to the faces of the flat by static charge but is safely dislodged when ultrasound is passed

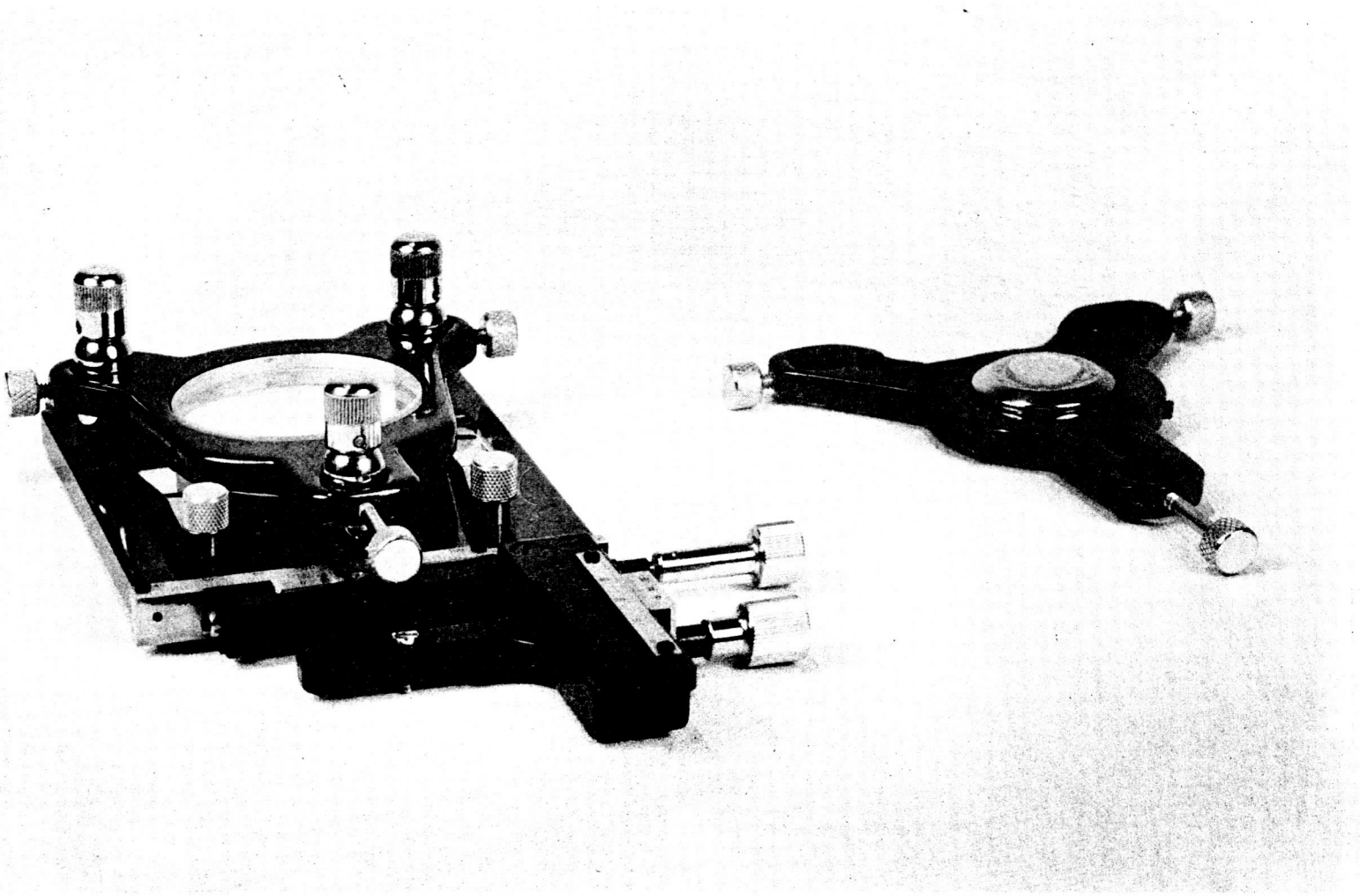

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Figure 11. Stage interferometer and attachment for small optical flat.

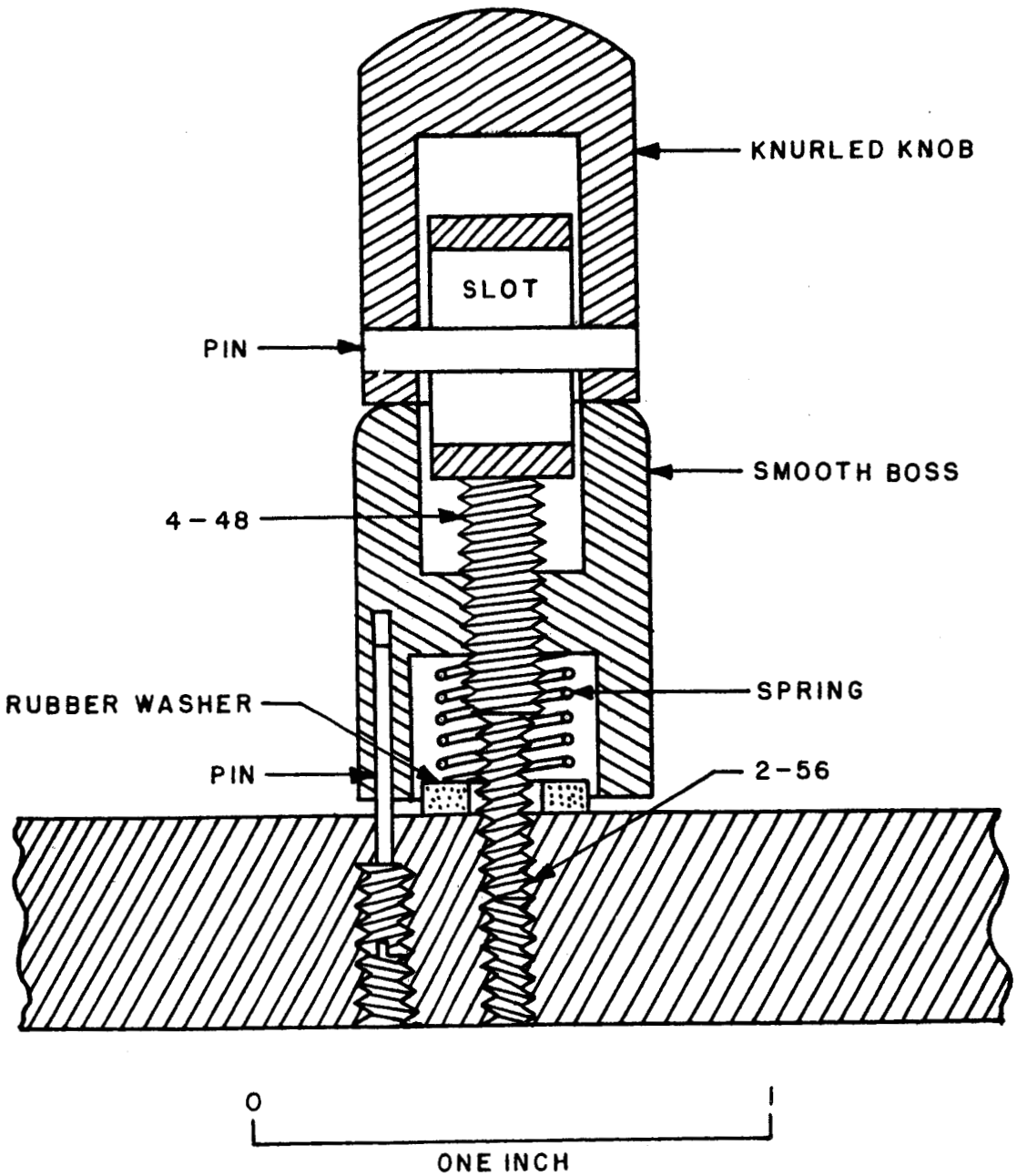

Figure 12. Cross section of differential adjusting screw shown 3X actual size.

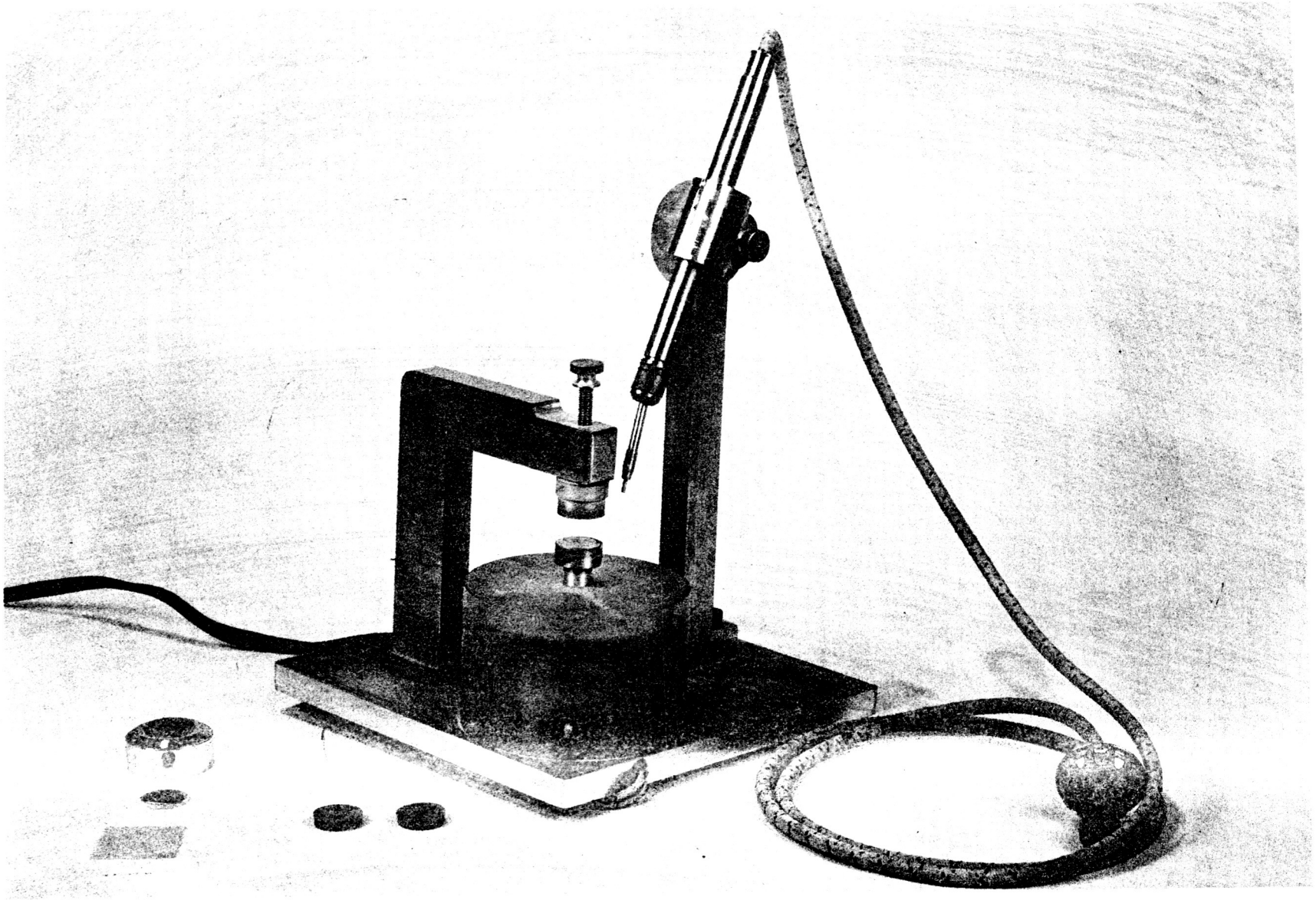

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Figure 13. Apparatus for cutting small optical flats.

through the methylene chloride. The finished flat is then waxed onto a chrome plated cup (fig. 11) and attached to an appropriate yoke for manipulation in the stage interferometer. Although such flats are capable of forming somewhat smoother interference fringes than mechanically polished flats, those of the former which we have made do not represent the ultimate in this regard that one might desire. Apparently even our elaborate cutting procedure produces some very superficial scratching of the dielectric coating although this does not appear to be serious. With this type of flat film thicknesses as small as 50 Å have been successfully measured. Figure 14 shows a set of interference fringes, made with such a flat, traversing the edge of a silicon monoxide film, 93 Å thick. The reproducibility of the measurement may be indicated by remarking that when two experimenters measured this step, one obtained a film thickness of 91.2 Å and the other 93.2 Å. These figures are averages derived from four fringes in the interferogram. The step was well delineated and in less favorable cases it will not always be possible to obtain this precision.

At this point, the multiple beam interferometer is largely a finished task. Since films thinner than the 50 Å one are in prospect, it might be profitable, however, to improve the flat-cutting process in regard to the removal of the cut flat from the cutter since this is where the scratching appears to occur. A more intriguing prospect is the adaptation of the new British float process glass to use as optical flat material. This glass, which is solidified against a molten tin surface during manufacture, combines a flatness approaching polished plate with a smoothness equal to that of firepolished sheet. Float process glass is not yet in production in the U.S.A.; however, we have been presented with a sheet from pilot plant production by one of the large glass manufacturers. This may be examined in the near future.

Optical hardware is being assembled that will allow the interferometer to form fringes of equal chromaticity, if the need for these should arise.

The ellipsometer ordered by HDL did not arrive until the end of July. In the meantime, work had been carried forward on one of the NBS instruments on a part-time basis. Over and above the complexities residing in this optical thin-film thickness measurement, unanticipated difficulties were encountered when vacuum-deposited thin films were examined. Especially with vacuum-deposited aluminum films, and to a lesser extent with vacuum-deposited gold films, a significant part of the reflected beam appeared to be depolarized. This effect was worse at large angles of incidence. With gold and at moderate angles of incidence, reasonable null balances could be obtained on the ellipsometer, depolarization notwithstanding. In experiments with aluminum oxide films grown on vacuum-deposited aluminum, the literature values for the optical constants of the base metal were of necessity used in the thickness computations. The results were absurd and it is painfully apparent that the optical constants of metallic aluminum are so sensitive to the conditions of preparation that a "best" literature value will not,

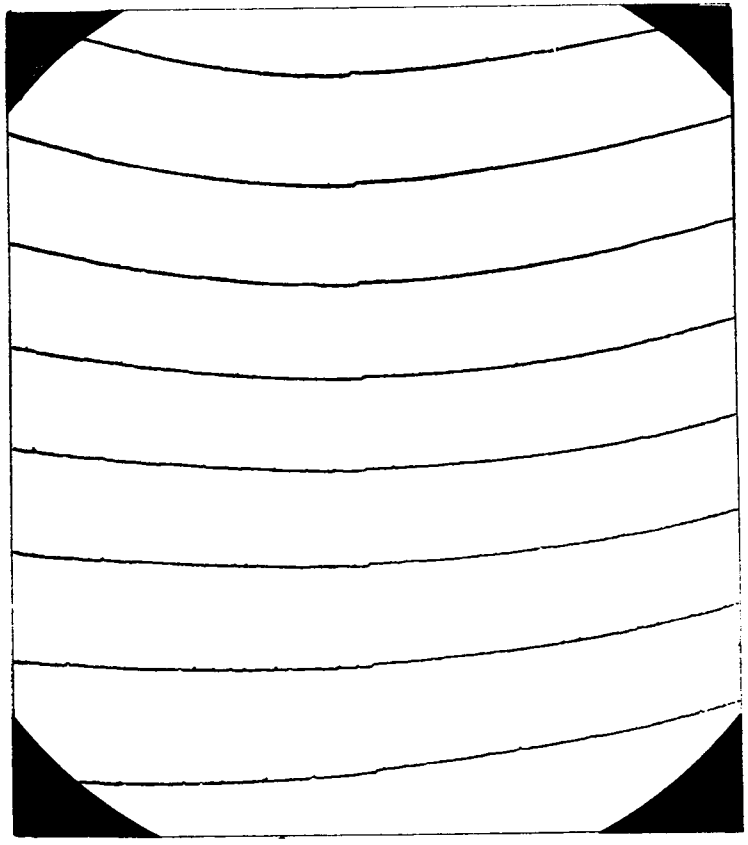

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Figure 14. Interference fringes crossing the edge of a silicon monoxide film 93Å thick.

in general, suffice. Emphasis was shifted to gold, where the optical constants of the metal film can be determined directly. Silicon monoxide was vacuum deposited over the gold. This system can be measured both by the ellipsometer and the interferometer. Here the values for the optical constants of the metal were reasonable although not every film agreed closely with the literature value. Even so the thicknesses of the silicon monoxide films calculated therewith were in serious disagreement with the interferometric values. The most logical explanation for this effect would be that the gold films were strained when first deposited and were partially annealed during the subsequent silicon monoxide deposition. Thus the optical constants measured for gold were no longer appropriate after the second deposition. Separate experiments on vacuum-deposited gold films alone showed that the optical properties do indeed change on heat treatment.

Clearly, further experiments on annealed gold films are indicated. Inasmuch as these would have been time consuming and inasmuch as the interferometric method now appeared to give the accuracy required for our work, the ellipsometer experiments were subordinated to the making of gold-silicon monoxide-gold tunneling or thermionic structures, wherein thicknesses were measured interferometrically.

During this period, a test chamber was assembled to enable the electrical characteristics of tunneling structures to be determined above and below room temperature, and in various atmospheres, or more particularly, in a vacuum. The chamber assembly consists of a bell jar, base plate, and a mechanical pump. The tubing for the evacuation of the chamber as well as that for the admission of gaseous ambients passes through the base plate along with electrical connections and the tubing for the circulation of refrigerant to the fixture to be described. The substrate on which a set of 12 diodes has been deposited is held in place on a copper heat sink by spring fingers. The temperature of the heat sink and diodes can be lowered by passing a refrigerant, usually liquid nitrogen, through the copper tubing provided. This tubing is formed into several close convolutions on the reverse side of the heat sink to which it is brazed. A thermocouple is provided to monitor the substrate temperature. Heaters can be incorporated to reduce the effect of the refrigerant at temperatures not far below room temperature or to heat the fixture above room temperature. Electrical connections to the diodes under test can be made by micromanipulator probes or a multiprobe connector. The former allow particular areas of a device to be probed. The latter is connected to an external switch box and permits all the diodes to be checked individually during a single pumping cycle. The test chamber is shown in figure 15. It has not yet been used in our experiments.

In the performance of the measurement of the electrical characteristics of the large number of thin-film diodes, extensive use was made of conventional transistor curve tracers because of their convenience and availability at HDL. Although they generally provide a measurement less elegant than the four-terminal method described earlier

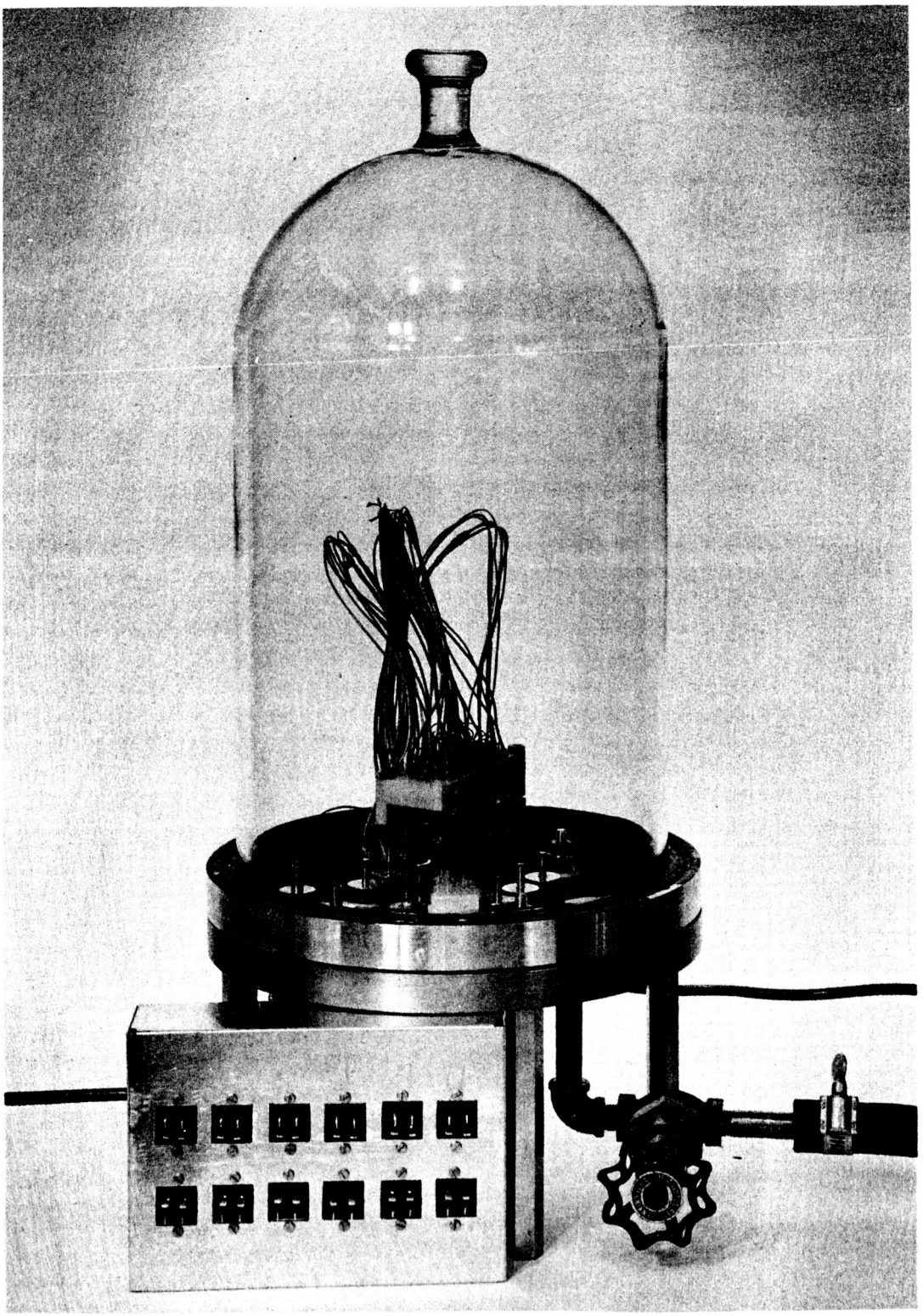

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Figure 15. Test chamber for arrays of thin-film diodes.

(ref 12), under carefully arranged conditions they can provide measurements that are basically as accurate as those from the four-terminal method. Under less carefully arranged conditions, there is indeed a sacrifice of accuracy. For example, some aluminum-aluminum oxide-aluminum diodes were recorded as apparently shorted (resistance of the order of the lead or metallic layer resistance) in the previous report (ref 13). In that measurement double contacts to each electrode of the diode (called emitter and base in this discussion in analogy with transistors) were tied together and the resistance determined from the current-voltage characteristic as seen on a transistor curve tracer. During this period, a closer look was taken at five closely similar diodes, also apparently short circuited using strictly four-terminal methods, i.e., by simultaneously measuring the direct current through one pair of emitter-base terminals of the diode and the open-circuit voltage across the other pair. Resistance values ranged from 0.045 to 5.65 ohms, as shown in table I. This evidently implies the existence of some residual barrier thickness or alternatively a finite number of filamentary shorts or metallic bridges in these diodes.

The necessity for accurate four-terminal methods, coupled with the time involved in point-by-point measurements on nonlinear diodes of limited stable lifetime, led to the development of an equivalent three-terminal method. This technique is particularly suited for use with a three-terminal input transistor curve tracer or two single-ended input amplifiers with a common ground. Briefly, one can show that three separate voltage measurements by this method at a constant current yield the voltage obtained from a four-terminal measurement at the same current level. This is seen from figure 16, which depicts the equivalent circuit for a metal-insulator-metal diode of unknown resistance, R, with double connections E_1, E_2 to the emitter electrode, and B_1, B_2 to the base electrode. The lead or contact resistances are conveniently represented in parallel, giving r_1 for the base when B_1 is shorted to B_2, and r_2 for the emitter when E_1 is shorted to E_2.

The method consists in alternately grounding the double contacts at one end and applying a constant current, I, into one of the double contacts at the other end, which determines the open-circuit potential at the remaining terminal. Thus a potential V_1 is measured with the circuit as shown in figure 16, and then with the diode turned end for end (reversing E and B) to determine V_2. Finally, both double contacts are shorted to yield V_3. The following equations ensue:

$$V_1 = (R + r_1)\ I \qquad (1)$$

$$V_2 = (R + r_2)\ I \qquad (2)$$

$$V_3 = (R + r_1 + r_2)\ I \qquad (3)$$

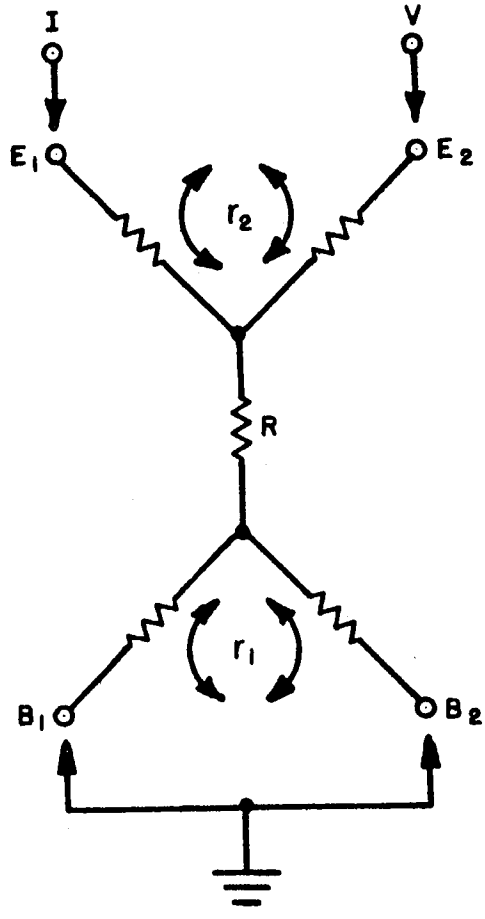

Figure 16. Equivalent circuit for three-terminal method. R represents unknown diode resistance. r_1, r_2 represent lead or contact resistances in parallel when each double connection B_1, B_2 and E_1, E_2 is shorted.

Table I. Detailed Examination of Apparent Shorts in Aluminum-Aluminum Oxide-Aluminum Diodes.

Diode No.	Crossover Area (mm^2)	Diode Resistance (Curve Tracer) R_{LV} (ohms)*	Diode Resistance (d-c 4-Terminal) R (ohms)**	Emitter Resistance (E_1-E_2) r_2 (ohms)	Base Resistance (B_1-B_2) r_1 (ohms)
D-1	4	12	.045	8	16
E-3	4	2.4	1.38	3	3
D-5	1	7.3	2.6	14.4	7.2
E-5	1	6	5.65	8.8	5.6
D-9	.25	14	2.52	30	12

* Resistance by transistor curve tracer at low voltage (< 100 mV) with E_1 shorted to E_2, B_1 shorted to B_2.

** Accurate resistance with direct current through E_1-B_1 and open-circuit voltage across E_2-B_2.

By inspection one sees that equation 3 subtracted from the sum of equations 1 and 2 gives the potential V over the unknown R in the equation:

$$V = V_1 + V_2 - V_3 = RI \qquad (4)$$

The equations obviously hold for any value of current, I, thus permitting point-by-point measurements where V is determined by three readings at each I value over a current range. It is also valid, however, to record by curve tracer or x-y plotter three curves of V_1, V_2, and V_3 versus I and by graphical addition and subtraction obtain the true I-V characteristic of a nonlinear diode. The switching circuit utilizing a transistor curve tracer shown in figure 17 was devised for this purpose. Note that V_3 is read before V_1 and V_2 for reasons not directly obvious, i.e., in practical structures the contact or lead resistances are all about the same value, making $r_1 \sim r_2$ and thus $V_1 \sim V_2$. Therefore, a comparison of V_1 with V_3 in the first <u>two</u> readings will sometimes eliminate the need for a third. For example, a perusal of equation 4 reveals that contact resistances are negligible at a given current level if V_1 (or V_2) $\sim V_3$. Similarly, a diode is probably shorted if V_1 (or V_2) $\sim$ 1/2 V_3. The third reading (V_2) is further complicated by the fact that here the diode, and, thus the current through it, is reversed. Therefore in measuring an asymmetrical or rectifying diode, the current, I, as well as S_2 must be reversed when reading V_2. This is easily accomplished by reversing the collector voltage in the curve tracer when taking this reading. Incidently, it should be noted that this method enables one to obtain the true I-V characteristics of transistors fabricated with double base connection, for example, by eliminating the ohmic voltage drop in the base layer and effectively contacting the active region.

3.5 Thin-Film Semiconductor Experiments

The new masks, described in the previous Progress Report, were used to deposit a matrix of 76 three-layer films onto a 3 by 1 in. glass slide. Since two such slides could be accommodated in the combined mask changer-substrate holder, 152 samples could be fabricated simultaneously.

With these new masks a number of aluminum-cadmium sulfide-gold depositions were made and the samples subsequently subjected to heat treatment under vacuum. These sandwiches are rectifying and are forward biased when the aluminum is positive with respect to the gold. Of samples heated in an oven with a 10°C temperature gradient (350° to 360°C) for 35 min, those at the 360°C end showed good rectification characteristics, but on continuing along the substrate toward the 350°C

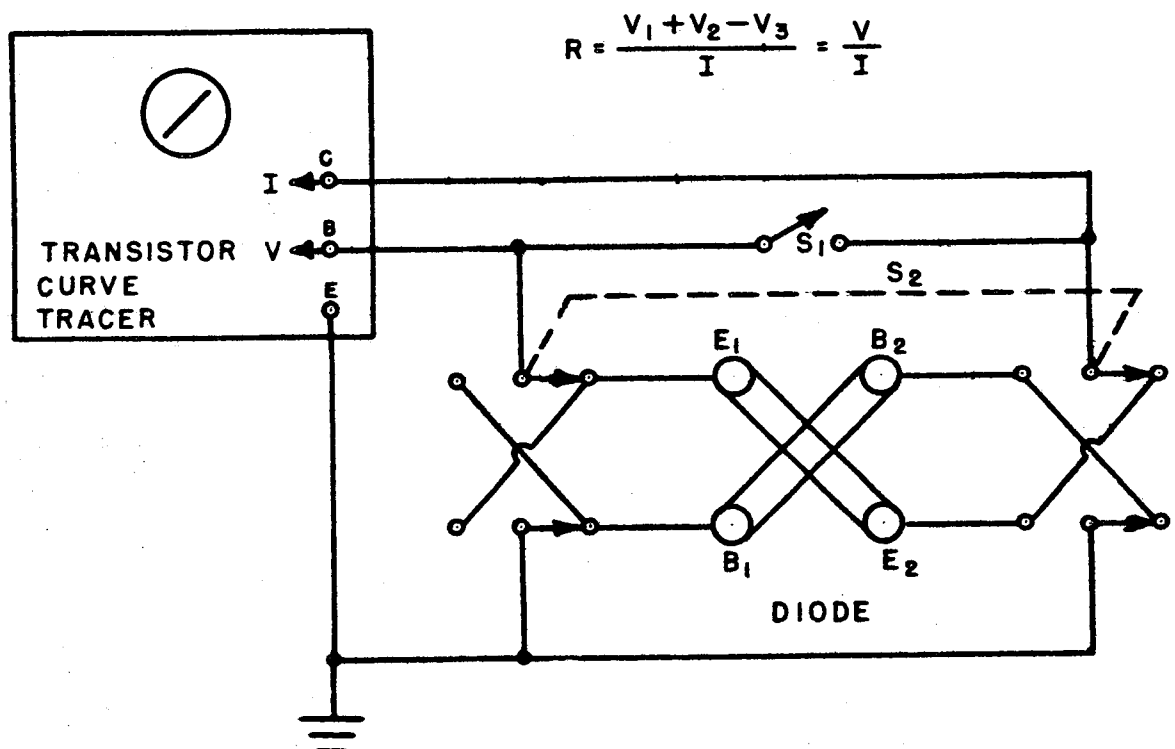

Figure 17. Three-terminal measurement utilizing a transistor curve tracer.

end, they showed a gradual degradation of the reverse characteristic. When similar structures were subjected to a similar heat treatment, but at a higher temperature (375° to 385° C), the best rectification characteristic was observed at the cooler end (375° C)--travel toward the hotter end (385° C) resulting in a diminution of the forward current. A good current-voltage characteristic can be obtained from every aluminum-cadmium sulfide-gold sandwich that is subjected to the proper heat treatment. A current-voltage curve for one of the diodes under discussion appears in figure 18. The sensitivity to heat treatment indicates that a compromise must be reached; too little heat will result in a low reverse impedance, while too much heat results in an increased forward resistance.

To study the resistivity of cadmium sulfide films, 24 depositions of the type gold-cadmium sulfide-gold were made, yielding 76 samples each time. Resistivities were calculated both before and after heat treatment. After heat treatment most of the resistivity values fell in the range 0.3 to 3.0 x 10^5 ohm-cm. Before heat treatment the value of resistivity was of the order of 0.1 to 1.0 ohm-cm and so an increase of about five orders has occurred during the heat treatment.

X-ray diffraction patterns of as-deposited and heat-treated cadmium sulfide films were made to determine if any free cadmium was present in the films. The X-ray analysis showed no detectable amount of free cadmium. Also, the films were highly oriented with the basal planes of hexagonal cadmium sulfide paralleled to the substrate surface.

The heated and nonheated cadmium sulfide films were studied by reflection electron diffraction. Both films gave the same diffraction patterns. The important conclusion is that no lines due to free cadmium were observed. The work summarized here will be reported in greater detail in a separate HDL technical report which is in preparation.

4. PROGRESS - ADMINISTRATIVE

4.1 Historical Survey

This section is in continuation of a policy to record highlights of papers published during the report period on thin-film radiation-resistant devices and associated experimental or theoretical studies, or materials technology. No slight by omission is intended in this selection from voluminous material. The emphasis during this period has been on supporting research aimed at understanding existing or proposed active devices rather than on the devices themselves. Thus, no new active triodes have appeared, but rather new embodiments or refinements of existing devices have been exploited. The greatest activity in triodes, as such, concerns the field effect or space-charge-limited type as opposed to the hot carrier type. Zuleeg of Hughes (Newport Beach) reports cadmium sulfide triodes (ref 14) with properties (for example: $g_m > 10.000$ μmhos) comparable to those of Weimer's (ref 15).

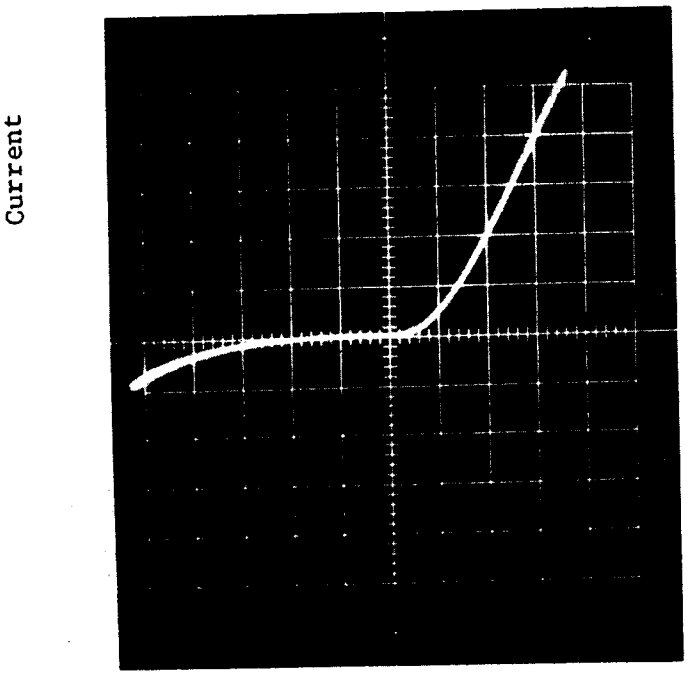

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Figure 18. Current-voltage characteristic of thin-film aluminum-cadmium sulfide - gold diode. Horizontal scale = 0.5 V/div.
Vertical scale = 0.5 ma/div.

He claims improved material control by molecular beam deposition from a Knudsen cell. Shallcross of RCA (Princeton) obtained similar results with cadmium selenide (ref 16). With colleagues, he also determined evaporated cadmium sulfide film properties (ref 17) such as mobility, trap density, and diffusion characteristics of copper and silver impurities. A variation of the triode with cadmium sulfide was described by Kauppila of General Motors (Dearborn) called the surface unipolar triodes (ref 18), which differs in that a highly doped surface layer is utilized for the active region. Latham, Lindholm, and coworkers (ref 19) developed new operational modes of the field effect triode using two gates, one for bias and the other for control. They also improved the design theory, accounting for the variable channel width from source to drain, in determining gate capacitance (ref 20). Richer and Middlebrook of California Institute of Technology published a series of papers on design theory for the field effect triode. One develops power law triode characteristics (ref 21). Another derives the characteristics by simplified charge control theory (ref 22). Several others consider basic limits of the derivations and properties of these devices (ref 23). A final paper (ref 24) refines the Lindholm et al (ref 20) calculation of input capacitance.

The principal noise at moderately high frequencies (megacycle region) in these triodes is ascribed by van der Ziel (ref 25) to be gate noise, i.e., input fluctuations coupled to channel region through the gate capacitance. Noise measurements by Bruncke (ref 26) agree with this theory.

Several papers involve refinements in the theory for space-charge-limited currents as the conduction mechanism in these devices. Lindmayer et al (ref 27) give a complete, normalized solution for the one-carrier model, so that the material constants are absorbed into dimensionless coordinates plotted as universal curves. A unified theory encompassing both carriers (electrons and holes) and traps was developed by Muller (ref 28). He also reported experimental work with gold blocking contacts on cadmium sulfide (ref 29), where emission at high fields fit Schottky theory.

Two additional papers on effects that might be encountered in cadmium sulfide triodes are worth noting. Chopra at Philco found evidence of avalanche injection in cadmium sulfide films (ref 30). Russ measured conductivity in cadmium sulfide and zinc sulfide with and without guard electrodes (ref 31) and thereby detected appreciable surface leakage currents.

As noted previously, little work was reported on experimental hot carrier triodes. The initial successes were difficult to reproduce, and some controversies arose concerning the mechanisms of operation (ref 32). This, therefore, stimulated many studies, both experimental and theoretical, of the separate mechanisms or parts of these devices, i.e., emitter or collector diode structures, base transport, barrier

heights for collection and emission, and the technology of the materials themselves. Atalla and Soshea (ref 33) made an analysis of these triodes and compared their performance with that of transistors. They show that the gain-bandwidth product, like that of the transistor, increases with current density. At a reasonable current density of 10^3 A/cm^2 the gain-bandwidth products, or maximum oscillating frequencies, are 60 gc for the metal base transistor, and 10 gc for the tunnel emission triode, compared with 5 to 12 gc for the bipolar transistor. Gossick (ref 34) developed the theory and design of one triode of this class, the metal-base transistor (ref 32) as well as the metal-semiconductor rectifier as a separate part of the device.

The bulk of the published work involved the study of diode structures. Pollack et al (ref 8, 35) found Schottky emission and a forming effect resulting from polarization in aluminum oxide diodes. This polarization, was attributed to migration of metallic ions to the negative electrode (cathode) thereby lowering the work function for emission in one direction. The effect was reversible in that conduction could be increased in the other direction when the diode polarity was reversed. Simmons and coworkers authored a series of theoretical and experimental papers, mainly supported by measurements on beryllium oxide diodes. The effect of dissimilar electrodes on conduction and breakdown was noted (ref 36). Rectification ratios varied with the metal used as counterelectrode; changes in breakdown voltage corresponded to differences in work function of the counterelectrode metal. Beryllium oxide diodes (ref 37) showed tunnel emission at low temperatures and tunnel-plus-Schottky thermionic-emission at room temperature, similar to results found in Emtage and Tantraporn's earlier work on aluminum oxide and polymer diodes (ref 38). The effect of potential barrier shapes on the tunneling current was explored, and a rectangular-barrier graphical approximation was evolved that fits data on beryllium oxide diodes (ref 39). A final paper (ref 40) developed a general expression for tunneling in symmetrical diodes, which is more tractible than Holm's equation at intermediate voltage (ref 41), but converges to Holm's simpler equations for low and high voltages, i.e., much less or greater than the barrier height. Geppert analyzed the effect of image forces, doping, space charge, and traps on the shape of the potential barrier for tunneling in the insulator region between metal electrodes (ref 42), which fit experimental data in the literature. Meyerhofer and Ochs (ref 43) obtained current-versus voltage data on aluminum oxide and beryllium oxide diodes, which fit rigorous tunneling theory. A postscript to this review of diode studies was added by Chow, who analyzed the errors involved with approximations used in the theoretical models of Holm and Stratton (ref 44). He also treated the effect of nonuniform thickness of the insulating film on the tunnel characteristic (ref 45).

Since base transport is an important consideration with hot carrier triodes as well as transistors (and little is known regarding the former), a series of papers was published on energetic electron

transport in metals. Stuart et al (ref 46) claim that ordinary diffusion theory is inadequate for describing this process and apply the more general Monte Carlo or random-walk method. A model was used in which excited electrons are inelastically scattered by electron-electron interactions. They show that the range or attenuation length measured through the film will generally be less than the calculated mean free path between electron-electron collisions, which is made up of zig-zag segments from random scattering events. Discrepancies between earlier theory and experiment are thereby resolved. Quinn, who developed the early theory based on the free electron gas approximation for a metal (ref 47), extended his description to that of a real metal with dielectric constant dependent upon energy band structure (ref 48). Combining these results with those of Stuart et al described above (which he also discussed at great length), a more consistent picture of electron range in metals is obtained, particularly of the anomalously low value reported for copper (ref 48). An additional refinement in the theory was made by Adler in reference 49, where he showed that solid state considerations such as the deviation from a spherical Fermi surface reduce the range of hot electrons in metals. This paper was also cited by Quinn to explain the low value in copper.

Several experimental papers on hot electrons in metals were reported. Collins and Davies (ref 50) measured the energy distribution of hot electrons in aluminum by retarding field measurements on an aluminum oxide tunnel cathode. White and Logan (ref 51) determined an attenuation length of 130 ± 40 Å for 4.7-eV electrons in gold by Schottky emission in reverse-biased gallium phosphide avalanche diodes.

The barrier height or work function, noted previously as an important parameter characterizing hot carrier triodes, was estimated in most of the diode or emission studies cited above. Many other works describe the determination of barrier height either indirectly from current-voltage or capacitance-voltage data, for example, or directly by photoelectric or other such measurements. They are too numerous to be discussed here, and some methods have been used only on bulk or single crystals, with but potential application to thin-film structures. The same remarks apply to a wealth of literature on materials technology, such as the preparation or properties of various thin films, some of which might contribute to the realization of a reliable active thin-film device.

Finally, several conferences during this period require some discussion. The Spring Meeting of the Electrochemical Society at Pittsburgh, Pa. (15-18 April) sponsored a Joint Symposium on Thin Films for Electronic Applications. Some of the papers were delivered by authors already cited above from the literature as amplification or extension of their work. Numerous others dealing with the technology of materials again have possible bearing on future thin film devices. A few are of more immediate interest and therefore deserve comments.

Miles and Smith of Arthur D. Little, Inc presented data on gaseous anodized aluminum oxide diodes which fit simple tunneling theory, showing negligible temperature or image force effects. Current was stable over ten decades to a maximum current density of 10^3 A/cm^2. Asymmetrical diodes, that is, with different counterelectrodes, were examined and showed rectification effects that correlated with literature values of the electrode work functions. Hickmott of General Electric (Schenectady) explored a voltage-controlled negative resistance in diodes of anodic aluminum, tantalum, zirconium, and titanium, in which the peak current occurred at a voltage proportional to the square root of the dielectric constant of the oxide. They exhibited square-law conduction (attributed to a possible space-charge-limited mechanism) up to this voltage and current. Operated in vacuum these diodes also emitted electrons in the negative resistance region, the threshold for emission occurring at a voltage somewhat less than half the vacuum work function, and an additional rise in emission at a voltage somewhat greater than the work function. Johnson of Westinghouse (Pittsburgh) discussed physical processes in cadmium sulfide field effect triodes. He showed that the transconductance is dependent upon the doping-mobility product rather than the mobility alone. He developed design theory for various cases, such as semiconducting cadmium sulfide with thin or thick gate insulator, and insulating cadmium sulfide. Results from the literature were cited to demonstrate that the highest transconductance is attained with semiconducting cadmium sulfide. Other effects, such as loss of gate voltage in filling surface-trapping states, and field ionization of shallow ionized donors, were considered as possible causes for departure from ideal characteristics and also were observed on devices fabricated in his laboratory.

The 1963 Solid State Device Research Conference held this year at East Lansing, Michigan (12 - 14 June) posted relatively few papers on thin-film device research. Weimer and coworkers at RCA (Princeton) presented a multielectrode refinement of their original cadmium sulfide triode. Miksic and colleagues from IBM (Yorktown Heights) discussed the behavior of cadmium sulfide transistors of their own design and construction. An amplification of his earlier theory, cited previously in this review (ref 25), on excess noise in field effect transistors was made by van der Ziel. Hershinger et al of Philco (Bluebell) disclosed a metal-base transistor utilizing graded zinc sulfide-cadmium sulfide films, the first polycrystalline thin-film version of this type device having power gain (approximately 20). Smith and Miles of Arthur D. Little, Inc elaborated on their work with aluminum oxide diodes (which was cited above in connection with the Electrochemical Society Conference at Pittsburgh). Finally Moll and colleagues at Stanford discussed their recent studies of hot electron energy and range in gold. Experimental data were obtained by both photoresponse of gallium arsenide-gold surface barrier diodes and emission from cathodes with a gold wedge film on silicon, which were consistent and in substantial agreement with that of other workers.

4.2 Contract Effort

Research programs at Philco Corporation and General Electric (Owensboro) were monitored by HDL for NASA. Work continued from June 1962 through September 1963. Detailed progress under these contracts is being reported separately; a resume of the work follows.

4.2.1 Philco Contract

The effort at Philco was applied in three areas:

(1) fabrication of MEA triodes,

(2) study of thin-film diodes, and

(3) analysis of the "back-scattering" problem.

The metal-edge-amplifier (MEA device) utilized two films of aluminum, separated by a thin film of oxide, placed side by side on a substrate of germanium. When the contract began, the Philco researchers were having trouble with electrical shorts in the oxide film, and were unable to make devices that they had previously made successfully. At first it was felt that an improvement in the vacuum would lead to more reproducible results; the change was made from a conventional oil-pump system to an ultra-high-vacuum system with an ion pump. Variations were made in oxygen pressure and substrate temperature to determine the effect on thermally grown oxide films. Finally the idea of thermal growth was abandoned, and two new methods of forming oxide films were studied. These methods were plasma anodization and reactive deposition. With the latter method it was eventually possible to reproducibly deposit very thin, compact, pinhole-free films. Moreover a servo system was assembled that could sense, and automatically control, the rate of deposition of the oxide film. In spite of this effort, the MEA devices were still not reproducible. An occasional device showed good characteristics, just as had been true at the beginning of the contract period. However most of the devices, by far, were not good. Reluctantly the experimenters decided that the real problem lay in the control of geometry in the structure, and that such control was beyond the limits of present technology.

The problem of back-scattering attracted early attention at Philco. It was felt that electrons passing from a metal into a semiconductor would be scattered by the potential step at the interface. Moreover a certain fraction of the electrons that had entered the semiconductor would suffer momentum-reversing collisions and be scattered out again. This was considered to be a serious factor that would limit the collection efficiency of any hot electron device. Consequently considerable effort was put into a theoretical analysis of this problem. Some interesting results were obtained and experiments were planned to verify some of the predictions of the theory.

It was the concern with back-scattering that led to the concept of the metal-oxide-semiconductor (MOS) diode. This was conceived to be essentially a metal semiconductor surface-barrier diode, but the oxide layer between metal and semiconductor was supposed to reduce back-scatter and enhance collection efficiency. MOS diodes of aluminum-aluminum oxide-germanium were fabricated in connection with the work on MEA devices already described. It was shown that a depletion region existed in the germanium, even when the oxide film was interposed (there had been some doubt about this point), but it was also shown that the presence of the oxide caused a "smearing out" of the potential gradient and caused a degradation of device characteristics. MOS diodes were also fabricated on cadmium sulfide (again using aluminum-aluminum oxide). At first the investigation centered around techniques for depositing cadmium sulfide and improving the quality of the film by appropriate heat treatment. Later some diodes were made and the current-voltage characteristics were recorded. It proved to be difficult to interpret these characteristics on the basis of either tunneling theory or conventional diode theory. The current was exponential with voltage, symmetrical with respect to polarity, and insensitive to changes in temperature.

It was found that visible light was emitted from these diodes when they were operated at 77°K with a strong d-c forward bias. The Philco investigators were interested in pursuing an investigation of this phenomenon, but were discouraged from doing so under the HDL contract.

HDL respresentatives urged Philco to concentrate on experiments that would prove or disprove the feasibility of the Mead triode. Accordingly Philco proposed an elaborate--but direct--experiment involving the shooting of low-energy electrons at a metal-insulator-metal sandwich. The experiment would measure attenuation of hot electrons in the first metal film and would determine whether back-scattering was a serious problem at the metal-insulator interface. The proposal was approved and preparations were undertaken to design and build a special electron gun, modify the vacuum system, and prepare suitable samples. These preparations were vigorously pursued and the experiment was on the verge of being performed at the time of expiration of the contract.

4.2.2 General Electric Contract

The purpose of the General Electric work was to experiment with materials and techniques in the hope of developing a practical tunnel cathode. The initial approach was to vacuum deposit strips of aluminum on a glass substrate, grow a thin aluminum oxide film by wet anodization techniques, and then vacuum deposit a counter-electrode of aluminum or gold. This method produced workable diodes from which measurable emission into the vacuum was obtained early in the contract period. No better system than this was ever found, although experiments

were conducted with a variety of other counter-electrode materials, substrate materials, and an alternative method of growing the oxide.

Aluminum and aluminum oxide were used in all diodes. Counterelectrode materials that were tried included nickel, aluminum, titanium, platinum, lead, magnesium, manganese, bismuth, tin, indium, and gold. No material proved to be as good as gold, presumably because no other material could be deposited in uniform films as thin as the gold films. This was substantiated by detailed examinations of the various films with an electron microscope.

There was also experimentation with the substrate material. There were two reasons for interest in the substrate: the smoothness of the substrate was important in determining the smoothness of the deposited films, and the heat conductivity of the substrate was important in determining the power levels at which burnout would occur. Experiments were performed with glass, ceramic, and metal substrates, but glass was the most practical.

Gaseous anodization was tried as an alternative method of growing the oxide film. It was felt that since this was an inherently cleaner process, it would have advantages over the method of wet anodization. However, the diodes produced by the newer technique were never as good as the ones produced by conventional methods. Another disappointment was the attempted use of barium oxide to lower the work function at the metal-vacuum interface. It was felt that depositing a thin film of barium on the gold electrode would enhance emission, but instead it produced electrical shorts and unreliable devices.

An important phase of the experimental work was concerned with the determination of optimum thicknesses for both the insulator and counterelectrode films. The best empirical values were about 100 Å for the aluminum oxide and 80 Å for the gold. G. E. oxide films of this thickness were capable of supporting a potential difference of 6 to 8 volts, and could pass direct current of density about 1 a/cm^2. Emission currents as high as several microamperes were obtained under d-c conditions from a 1-mm^2 electrode. Most measurements were conducted under pulse conditions (with about a 10% duty cycle) in order to avoid thermal destruction of the devices. The best results obtained were as follows. Diodes could be made reproducibly that would, for a period of several days, operate at peak currents of 0.42 amp and emit 1.4 ma (60 ma/cm^2) into vacuum. By the end of the contract period, it had been shown that there were good reasons for believing that a practical cold cathode could eventually be built, at least for low current applications.

5. CONCLUSION

At the inception of the work on Radiation-Resistant Electronics at HDL, the object was to determine the feasibility of a thin-film,

hot electron triode of the type proposed by Mead. It was felt that such research would not provide an immediate solution to any existing problem, but might be the basis for a long range attack on the problem of what to do about radiation damage to transistors in space environments. In addition to radiation-resistance, the Mead device offered the advantages of small volume, low power consumption, and the capability of being fabricated from polycrystalline materials.

At the outset, the principal questions were:

(1) whether tunneling was a practical mechanism for use in a hot electron emitter,

(2) whether hot electrons could propagate useful distances through metal films, and

(3) whether a collector could be realized that would be sufficiently opaque to cold electrons and transparent to hot electrons.

Concerning the first of these questions, there are indications that a practical tunnel emitter is in the offing. It has been shown that films of the required thinness can be made, and that hot electron currents can be produced. For example, hot electrons have been extracted from cold cathodes in at least three research programs, viz: Westinghouse, General Electric,and Raytheon. It has been difficult to establish that tunneling was actually the basic mechanism, but even here the evidence is mounting. Data, such as that produced by Miles and Smith, offer convincing evidence that tunneling is the dominant current mechanism in some thin-film structures.

At first the preparation of films thin enough for tunneling went beyond what was state-of-the-art. Whereas optical films (1/4 wavelength of visible light) had previously been considered thin at 1000 Å, tunneling films had to be an order of magnitude thinner, i.e. less than 100 Å. Progress has been made in the past two years in achieving higher vacua, refining the processes of control and measurement, and developing new techniques such as gaseous anodization. Because of this progress, it is now possible to fabricate pinhole-free films so thin that tunneling should definitely occur.

Any uncertainty remaining about tunneling arises from two factors: complications that have made it difficult to interpret the data, and the necessity for distinguishing tunneling from other processes that can occur simultaneously. Examples of the kinds of complications countered are: (1) aging effects, where the properties of a sandwich would vary in time before stabilizing, (2) field effects, where the applied voltage would cause irreversible changes such as ion diffusion, and (3) electrical instabilities, where sandwiches would burn through or breakdown and change suddenly from one stable I-V characteristic to another. There has been steady progress in understanding these phenomena,

and improving the techniques of film fabrication. One example is the steady improvement in quality of the vacuum-deposited SiO films under the HDL contract.

Mechanisms other than tunneling that can provide current in a thin-film sandwich include thermionic emission, injection through pinholes, and space-charge-limited current. These processes can be distinguished from each other, but this requires extensive experimental data, which is still being collected. Not enough current-voltage characteristics have been obtained as yet, at different temperatures, on samples with known parameters (such as film thickness). It is felt that, with a moderate amount of additional research, a practical emitter for the solid-state triode will soon be established.

The second question was concerned with the propagation distance of hot electrons. Fortunately, as a result of work done at several laboratories, it has been possible to get an affirmative answer to this question. Recent developments in Quinn's theory by Stuart, Wooten, and Spicer have already been mentioned. Both theoretically and experimentally, it has been shown that electrons, with energy a few volts above the Fermi level, can travel distances of several hundred angstrom units through metal films without appreciable loss of energy. This removes what was once an objection to all hot electron devices.

The third question, related to the collection of hot electrons, has received the least attention and is still largely unanswered. This is the area where future research will have to be directed. The only collectors built so far have used the surface barrier that occurs at a metal-semiconductor interface. The original metal-interface-amplifiers (MIA) used single crystal germanium. Recently, at Philco, metal-base-transistors have been made in which collection was accomplished in a polycrystalline film, made of mixed sulfides of zinc and cadmium. The collection efficiency of all these devices has been low. From consideration of the back-scattering problem, it has been theorized that the collection efficiency of any hot electron device, similar to the Mead triode, will be low. The theory has not been confirmed; some experiments were planned, but have not yet been carried out. It will be important to determine from such experiments whether or not collection efficiency will actually impose any serious limitation on hot electron triodes.

A brief discussion of the comparison of different kinds of thin-film devices and the analysis of Atalla and Soshea have been mentioned. In comparisons of this kind, the tunnel triode comes off badly because tunneling requires a thin insulator, and this results in a large input capacitance. It has been stated that, because of this, the tunnel triode would always have poorer frequency performance than nontunneling devices. While this has not been firmly established (since frequency cutoff depends on more than emitter capacitance), it represents a limited point of view even if true. The reason for interest in the Mead

triode was radiation resistance. Frequency performance was of secondary interest. The Mead device still offers the possibility of being a radiation resistant substitute for conventional transistors. It would be of considerable value as such even if it should prove to have lower α and somewhat lower frequency cutoff than conventional transistors. This is why it is still worthwhile to continue the investigation of the Mead triode. It is felt that research into hot electron phenomena has proceeded satisfactorily, but it is still too early to assess the ultimate capabilities and limitations of the devices.

Concurrent with the study of the Mead triode, there has been continued interest in other hot electron devices such as the metal interface amplifier (MIA) and the metal base transistor (MBT). Also there has been interest in devices that are not hot electron, but are still thin-film, such as the metal-edge-amplifier (MEA) and the space-charge-limited triode. A brief review of the status of these devices will be presented.

Originally it was felt at Harry Diamond Laboratories that the MIA was of little interest for radiation-resistant electronics because it depended on the use of a single crystal semiconductor for collecting the hot electrons. Similarly the MBT, in its original form, used single crystal material. The major claim for the MBT was its predicted high maximum frequency of operation. The claim was also made that the MBT, being a majority carrier device, would be more radiation-resistant than conventional transistors. (This argument would also hold for the MIA.) However, it is not clear what fraction of the collector-base current in an MBT would be made up of minority carriers from the semiconductor. Radiation decreases minority carrier lifetime and increases I_{CO}. It is felt that the Mead triode, with no semiconductor would still offer the best possibility of a radiation-resistant substitute for transistors.

There is one development that could alter this conclusion. The work at Philco Corporation showed that a surface barrier exists in polycrystalline as well as in single-crystal semiconductor material. This opens up new opportunities. It means possibly that both the MIA and the MBT can be built in a form that will be more radiation-resistant than was previously thought possible.

The MEA was discussed in the section on external effort. Success here seems to depend on the development of some new method for achieving control of electrode spacing. As with other devices, the radiation resistance of the MEA depends on whether it can be built on polycrystalline material.

The space-charge-limited triodes of Wright and Weimer were also discussed. The field effect version, in particular, seems to show much promise. Triodes have been built that exhibited power gain and good device characteristics, the major problem being lack of reproducibility.

In estimating radiation sensitivity, one can say that the device is a majority-carrier device that works in polycrystalline material. These factors favor radiation resistance. On the other hand, the space-charge-current mechanism is (in theory) very sensitive to trap density; from this point of view, the device looks less resistant. No definite answer can be given at this time.

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